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ихайлович Ча́плин (28 июля (9 августа)
0 ноября 1931) — русский и советский учёный
пециалист в области отопительно-
ионной техники.

Дед писательницы Веры Чаплиной[2].
Потомственный дворянин из рода Чаплиных,
восходящего[3] ко Льву Чаплину (середина XV века)[4].

В 1883 году окончил Императорское Московское техническое училище (ИМТУ), а в 1898 году получил в училище должность профессора. В 1898—1909 гг. также преподавал в Московском Училище живописи, ваяния и зодчества.

В 1895 году вместе с коллегой по ИМТУ архитектором В. Г. Залесским Чаплин организовал торговый дом «В. Залесский и В. Чаплин». В 1902 году Залесский и Чаплин построили собственный дом на Большой Дмитровке, 16, в котором их семьи имели по отдельному этажу и где располагались помещения их технической конторы[5]. Среди реализованных проектов торгового дома «В. Залесский и В. Чаплин» — системы отопления и вентиляции почти 1500 зданий[6] в десятках городов Российской Империи: Музей изящных искусств имени императора Александра III, Румянцевский музей, Политехнический музей, Аудиторный корпус Московского университета, Императорское Московское техническое училище, Московское училище живописи, ваяния и зодчества, Александровское военное училище, Брестский (Белорусский) вокзал, автозавод А. М. О., Сандуновские бани, гостиница Метрополь, ресторан Эрмитаж, здание страхового общества «Россия», Алексеевская глазная больница, странноприимный дом Шереметева, храм Василия Блаженного, склеп великого князя Сергея Александровича в Чудовом монастыре, Перервинский монастырь, Заиконоспасский монастырь, храм Марфо-Мариинской обители — все в Москве; храм-усыпальница Юсуповых в Архангельском, Петербургская городская детская больница, Путиловский завод, Архангельские городские ряды, Нижегородский городской театр, Киевский коммерческий институт, царский дворец в Ливадии, Иркутский завод «Треугольник», детская больница в Баку, фабрика Столыпина в Пензе, храм Казанской Божией матери в Риге, электрическая станция в Рязани, Тамбовский пороховой завод, Волжско-Камский банк в Самаре, Калужское Епархиальное училище, Императорский Харьковский университет и многие другие.

В 1903 году В. М. Чаплин создал первую [illegible] водяного отопления с побудителем [illegible]. Предложенная им водо-водяная [illegible] прототипом широко [illegible] систем отопления [illegible] теплофикационных [illegible]

165 ЛЕТ СО ДНЯ РОЖДЕНИЯ
9 августа 2026 г.

ЧАПЛИН ВЛАДИМИР МИХАЙЛОВИЧ 1861-1931 гг.

РУССКИЙ УЧЁНЫЙ, СПЕЦИАЛИСТ В ОБЛАСТИ ОТОПИТЕЛЬНО-ВЕНТИЛЯЦИОННОЙ ТЕХНИКИ

Г. МОСКВА

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Study of rheological properties of thermostable paints obtained by modification of silica sol

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Исследование реологических свойств термостабильных красок, полученных путем модификации золя кремнезема

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Abstract

This study investigates the rheological properties of heat-resistant composite paints modified with a synthesized organosilicon modifier based on silica sol and liquid glass. The modifier was prepared by the chemical modification of silica sol with an organic component and incorporated into paint formulations at different concentrations. The effects of modifier content on the viscosity and density of the coatings were evaluated using standardized testing methods in accordance with GOST and ASTM requirements. The results demonstrated that increasing the modifier concentration gradually increased the viscosity of the paint systems due to the formation of a stronger three-dimensional network between the modifier molecules and colloidal silica particles. At the same time, the coating density slightly decreased because of the lower density of the organic modifier, while the structural integrity of the coatings was maintained. Comparative analysis showed that paints containing 4–6 wt.% modifier exhibited the best balance between rheological behavior, storage stability, and application properties. The synthesized modifier improved the processing characteristics and is expected to enhance the thermal performance of the coatings. These findings demonstrate that the developed modifier is a promising additive for producing high-performance heat-resistant paint systems with improved rheological properties and potential applications in protective coatings for industrial and construction materials.

Аннотация

В данном исследовании изучаются реологические свойства термостойких композитных красок, модифицированных синтезированным кремнийорганическим модификатором на основе золя диоксида кремния и жидкого стекла. Модификатор был получен путем химической модификации золя диоксида кремния органическим компонентом и введен в состав красок в различных концентрациях. Влияние содержания модификатора на вязкость и плотность покрытий оценивалось с использованием стандартизированных методов испытаний в соответствии с требованиями ГОСТ и ASTM. Результаты показали, что увеличение концентрации модификатора постепенно повышает вязкость лакокрасочных систем за счет образования более прочной трехмерной сетки между молекулами модификатора и коллоидными частицами диоксида кремния. В то же время плотность покрытия незначительно снижается из-за меньшей плотности органического модификатора, при этом структурная целостность покрытий сохраняется. Сравнительный анализ показал, что краски, содержащие 4–6 мас.% модификатора, демонстрируют наилучший баланс между реологическим поведением, стабильностью при хранении и свойствами нанесения. Синтезированный модификатор улучшает технологические характеристики и, как ожидается, повысит тепловые свойства покрытий. Полученные результаты демонстрируют, что разработанный модификатор является перспективной добавкой для производства высокоэффективных термостойких лакокрасочных систем с улучшенными реологическими свойствами и потенциальным применением в защитных покрытиях для промышленных и строительных материалов.

Keywords: heat-resistant paint, silica sol, liquid glass, organic modifier, rheological properties, viscosity, density, thermal stability.

Ключевые слова: Термостойкая краска, золь диоксида кремния, жидкое стекло, органический модификатор, реологические свойства, вязкость, плотность, термическая стабильность.

Introduction

Today, the construction and finishing materials market is extremely diverse. High competition in this field drives the search for new, technologically advanced solutions to meet consumer needs[1,pp.1 – 2].

Ease of use and process speed play an important role in construction. Today, coatings containing silicate groups rightfully hold a leading position in terms of corrosion protection and thermal stability. Combining high efficiency, quality, and application speed, these monolithic bonding coatings have proven themselves to be heat-resistant, waterproof, protective, and anti-corrosion materials[2,pp.3 – 4].

The main obstacle to the widespread adoption of this type of material is the difficulty of applying them. The need for complex and expensive equipment and skilled workers, along with the low lightfastness of most systems, significantly limit the use of this type of coating[3,pp5-6].

Thermally and photostable coatings in the form of monomolecular (one molecule thick) layers or thin films are produced by treating materials with solutions, emulsions, or (less commonly) double water—substances that interact poorly with water but adhere strongly to surfaces.

The existing multi-stage technology for producing and using thermally stable polymers has led to lengthy synthesis, reduced yields of the target product (especially under laboratory conditions), and hinders the transition to a continuous process.

Experimental industrial process control provides the most optimal process flowsheet for their production using hetero functional condensation and hydrolysis, which simplifies the technology and reduces labor costs, energy, and material consumption[4,pp.4 – 5].

Acrylic acid, silica sol, liquid glass, and ethylene glycol were used as fillers during the synthesis process using the technology for developing thermally stable polymer composites using the developed method. In the Republic of Uzbekistan, large-scale measures are being taken to ensure fire safety of buildings and structures by creating and improving modern fire-fighting equipment and systems.

In this area, in particular, a number of studies are being conducted to create fire-resistant building materials based on local raw materials and to improve the quality of fire extinguishing methods and technical equipment[5,p.1].

Today, scientific research is being conducted worldwide to develop new, environmentally friendly, and cost-effective methods for synthesizing highly effective fire-resistant expanding polymer composite coatings from inexpensive raw materials and their application in the oil and gas, chemical, medical, and various construction industries[6,p.2].

Since the 20th century, SiO_2 nanoparticles have been widely used in many products, and since the 1960s, metallic nanoparticles have been used. It has been established that compacts of these very small (~10 nm) crystals possess unique physical properties that can be exploited for various engineering purposes[7,pp.8 – 9].

Many applications of nanocomposites are based on the properties of individual nanoparticles (sensors, medical diagnostics, homogeneous catalysis, etc.). At the same time, there are important areas requiring self-assembling nanoparticle composites (nanoelectronics, optoelectronics, photonics, heterogeneous catalysis, etc.).

Currently, much attention is being paid worldwide to the development of fire protection products based on modern technologies and their use to improve the fire resistance of building structures and materials. Accordingly, targeted scientific research in areas such as creating models of the mechanisms of physico-thermochemical processes occurring in protective coatings under the influence of heat; developing compact, accurate, and rapid methods for assessing the impact of fire-retardant fillers on the fire safety of building structures and materials; and creating a new generation of highly effective heat- and fire-protective coatings based on readily available natural resources is important. Conducting scientific research in the above-mentioned areas confirms the relevance of the topic of this dissertation[8,pp.1 – 2].

Materials are evaluated for their heat resistance, fire resistance, and resistance to sunlight and heat during the summer months. Thermal conductivity, flame resistance, and other factors are important thermal insulation properties, chemical stability of suspensions, and performance characteristics that influence the quality of the material.

In turn, these properties are achieved through the presence of silicate groups in the material and metal oxides in the paint. Studying these properties allowed us to investigate the thermal degradation of paint and varnish compositions on wood and rheological properties such as viscosity, adhesion, and density[9,pp.3 – 5].

Research object and methods

The study subjects were heat-resistant modifiers obtained by modifying liquid glass and silica sol, as well as paint samples containing modifiers in various percentage ratios. The study was conducted in accordance with the requirements of GOST 8420 – 74 and GOST 11066-74 for studying the rheological properties of paints and varnishes, as well as using product analysis methods compliant with international standards (ASTM).

Research results

In laboratory studies, an organic modifier obtained by reacting acrylic acid with ethylene glycol and silica sol were reacted in various weight ratios. A total of four different samples were prepared, and the resulting heat-resistant modifier was added to gray and red paints at a rate of 4 % by weight. The viscosity of the resulting paints was studied in accordance with the requirements of GOST 8420 – 74 (Table 1)[10,pp.1 – 3].

Table 1.

The influence of the concentration of colloidal silicon dioxide and modifier on the viscosity of the system

№	Quantity of components (g) in mass ratio		Type of pigment	Paint KO 8101 amount kg	Viscosity [VZ-246 (Ø4 mm)(s)]
	Silica sol	Modifier			
1	95	5	Red pigment (Iron Oxide Red 130)	2.5	53.3

№	Quantity of components (g) in mass ratio		Type of pigment	Paint KO 8101 amount kg	Viscosity [VZ-246 (Ø4 mm)(s)]
	Silica sol	Modifier			
2	90	10		2.5	54.3
3	85	15		2.5	56.7
4	80	20		2.5	59.1
5	95	5	Silver pigment (Iron Oxide Black 750 and KRONOS 2190 mixture)	2.5	55.1
6	90	10		2.5	56.3
7	85	15		2.5	58.2
8	80	20		2.5	63.9

Table 1 shows how the viscosity of a heat-resistant paint and varnish composition changes as a result of varying the mass ratio of colloidal silica to modifier. In the experiments, the paint volume for all samples was assumed to be the same—2.5 kg—and the viscosity was determined using a VZ-246 viscometer (Ø4 mm).

The table results show that as the modifier content increased from 5 % to 20 %, the systems viscosity steadily increased. This is explained by the formation of a spatial lattice structure due to the interaction of modifier molecules with colloidal silica particles and an increase in the internal resistance of the system.

For compositions using red pigment (iron oxide red 130), the viscosity was 53.3, 54.3, 56.7, and 59.1 s, respectively. With an increase in modifier content from 5 % to 20 %, viscosity increased by 5.8 s, or 10.9 %. Specifically, a significant increase in viscosity was observed in the 15–20 % modifier range, indicating the formation of stronger structural bonds in the composition.

A similar trend was observed in systems using gray pigment (a mixture of black iron oxide 750 and KRONOS 2190). Viscosity values were 55.1; 56.3; 58.2; and 63.9 s, respectively. The difference between the initial and final values was 8.8 s, or 16.0 %, indicating a stronger effect of the modifier on viscosity in the gray pigment composition.

When comparing the two pigment types, the viscosity of the gray pigment-based compositions was higher than that of the red pigment sample at all concentrations. The difference was 1.8 s at 5 % modifier, 2.0 s at 10 %, 1.5 s at 15 %, and 4.8 s at 20 %. In particular, the sharp increase in viscosity in the gray pigment system at 20 % modifier concentration is explained by the high dispersion of these pigment particles and their stronger physicochemical interaction with the modifier.

Table 2.

The influence of modifier concentration on the viscosity (at 20°C) of the obtained TB-1 (heat-stabilized paint)

	The amount of modifier in the paint %	Ø2 mm nozzle(s)	Ø4 mm nozzle(s)	Ø6 mm nozzle(s)
Plain white enamel sample KO-811	0	102.1	56.5	35.3
Paint containing gray pigment KO-8101	0	107.6	57.2	39.0
TB-1	2	109.3	59.4	41.7
TB-1	4	112.7	60.2	45.2

	The amount of modifier in the paint %	Ø2 mm nozzle(s)	Ø4 mm nozzle(s)	Ø6 mm nozzle(s)
TB-1	6	114.4	63.3	47.4
TB-1	8	117.9	65.2	49.1
TB-1	10	119.7	71.1	52.3

The effect of modifier concentration in the heat-resistant paint TB-1, presented in Table 2, on viscosity at 20°C was studied using viscometers with nozzles of various diameters (Ø2, Ø4, and Ø6 mm). For comparison, widely used industrial paints KO-811 with white enamel and KO-8101 with gray pigment were used as control samples.

The table results show that the viscosity of the control samples with a Ø2 mm nozzle ranged from 102.1 to 107.6 s, with a Ø4 mm nozzle from 56.5 to 57.2 s, and with a Ø6 mm nozzle from 35.3 to 39.0 s. These values represent standard rheological properties for industrial paints. When the modifier was introduced into the TB-1 composition, the viscosity gradually increased for all nozzle diameters. At a modifier content of 2 %, the viscosity was 109.3 s for a Ø2 mm nozzle, 59.4 s for a Ø4 mm nozzle and 41.7 s for a Ø6 mm nozzle. With an increase in the modifier content to 10 %, these values reached 119.7 s, 71.1 s and 52.3 s, respectively. The results show that with an increase in the modifier content from 2 % to 10 %, the viscosity increased :from 109.3 s to 119.7 s for a Ø2 mm nozzle, i.e. by 10.4 s (9.5 %); From 59.4 s to 71.1 s for a 4 mm nozzle, an increase of 11.7 s (19.7 %); in a 6 mm nozzle, the time increased from 41.7 s to 52.3 s, an increase of 10.6 s (25.4 %).

Furthermore, the increase in viscosity with increasing modifier content is nearly linear, indicating that the modifier molecules form a strong spatial network with colloidal silicon dioxide and binder components. This results in increased structural integrity of the paint, reduced pigment particle deposition, and improved coating formation.

When comparing TB-1 samples with KO-811 and KO-8101 industrial paints, a clear increase in viscosity is observed as a result of the modifiers use. In particular, when using 8–10 % modifier, the viscosity of TB-1 is significantly higher than that of control samples, confirming its high rheological stability[11,pp.5 – 8].

Overall, the experimental results showed that with increasing modifier concentration in TB-1, the paints viscosity increases and its processing properties improve. A modifier content of 4–6 % provides an optimal balance between paint wear resistance, storage stability, and coating properties, while a modifier content of 10 % provides the highest viscosity, but ease of application in industrial settings must also be considered. These results confirm that the modifier plays an important role in optimizing the composition of TB-1 heat-resistant paint.

Table 3.

Change in density of enamel paint with different concentrations of modifier (at 20°C)

% heat-resistant modifier in KO 8101 paint	Empty pycnometer weight, g.	Mass of the pycnometer with the test substance, g.	Pycnometer volume at test temperature, ml.	Sample density g/ sm^3
0	20	51	20	1,3 g/sm^3
2	20	50.94	20	1,297 g/sm^3
4	20	50.90	20	1,295 g/sm^3
6	20	50.78	20	1.289 g/sm^3

% heat-resistant modifier in KO 8101 paint	Empty pycnometer weight, g.	Mass of the pycnometer with the test substance, g.	Pycnometer volume at test temperature, ml.	Sample density g/ sm³
8	20	50.74	20	1.287 g/sm³
10	20	50.70	20	1.285 g/sm³

Based on the data presented in Table 3, it can be concluded that changes in the coating density as a result of increasing the amount of heat-resistant organic modifier in the paint composition were determined using the pycnometric method. In the experiments, the mass of the empty pycnometer (20 g) and the volume of the pycnometer at the test temperature (20 ml) remained constant, and the amount of modifier in the paint composition varied in the range of 0–10 %. This allows us to accurately estimate the dependence of the obtained results on the amount of modifier.

According to the results, the density of the control sample without the addition of modifier was 1300 g/sm³. With an increase in the modifier content to 2 %, the density decreased to 1.297 g/ sm³, at 4 % — to 1.295 g/sm³, at 6 % — to 1.289 g/sm³, at 8 % — to 1.287 g/sm³, and at 10 % modifier — to 1.285 g/sm³. Overall, increasing the modifier content from 0 % to 10 % decreased paint density by 0.015 g/sm³, or approximately 1.15 %.

The mass of the test substance in the pycnometer also gradually decreased with increasing modifier content. Specifically, this value decreased from 51.00 g to 50.70 g. This indicates that the modifier has a relatively low-density organic structure and reduces the average density of the paint and varnish composition.

The decrease in density is explained by the penetration of modifier molecules into the silicate binder phase and a slight increase in free volume in the composition. Moreover, the presence of carbon chains and functional groups in the organic modifier creates a lower density compared to the inorganic binder phase. As a result, its bulk density decreases slightly without reducing the overall mass of the composition.

This change is considered positive from a practical perspective, as a slight reduction in coating density improves paint flow, reduces coating mass, and optimizes material consumption when coating large surfaces. Moreover, the very small decrease in density (1.15 %) means that the structural integrity of the composition is maintained, and the addition of the modifier does not negatively affect the mechanical stability of the coating.

These results are also consistent with the adhesion and heat resistance results obtained in previous experiments. That is, although paint density decreases slightly with increasing modifier content, its adhesion and heat resistance improve significantly. This confirms that the synthesized organic modifier is capable of improving performance while simultaneously lightening the paint composition[11,pp.1 – 5].

Conclusion

The present study demonstrated the successful preparation of a heat-stable modifier by modifying silica sol with a synthesized organic modifier and confirmed its applicability in the formulation of heat-resistant paint coatings. Rheological investigations showed that the composition of the modifier significantly affects the technological properties of the coating[12,pp.4 – 7].

The optimum silica sol-to-organic modifier mass ratio was determined to be 4:1, providing the most favorable rheological characteristics. Under these conditions, the viscosity reached 59.1 s for the red coating and 63.9 s for the gray coating. The gray coating based on pigment K 8101 exhibited higher viscosity, density, and adhesion, making it the most suitable formulation for heat-resistant applications.

The scientific novelty of this work lies in establishing the relationship between the composition of the silica sol-based modifier and the rheological behavior of heat-resistant coatings, as well as identifying an optimal formulation that enhances coating performance. The synthesized modifier improves viscosity, structural stability, adhesion, and consequently the thermal stability of the coating.

Compared with commercially available heat-resistant coatings such as KO-8101, KO-811, KO-814, Elcon, and Certa, the developed silica sol-modified coating offers a water-based silicate system with improved rheological stability and strong adhesion while employing a relatively simple synthesis route based on readily available raw materials. Unlike conventional silicone-organic coatings, which generally require organic solvents, the proposed formulation provides an environmentally friendlier alternative while maintaining the characteristics required for heat-resistant protective coatings[13,pp.11 – 13].

Therefore, the developed modifier represents a promising material for manufacturing heat-resistant coatings intended for metal structures, industrial furnaces, pipelines, exhaust systems, and other equipment operating at elevated temperatures[14,p.2]. The obtained results provide a scientific basis for further optimization and industrial application of silica sol-modified heat-resistant coating systems.

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Mapping the knowledge structure and research evolution of ammonium nitrate thermal stability: a bibliometric review

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Анализ структуры знаний и эволюции исследований термической стабильности аммиачной селитры: библиометрический обзор

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Abstract

The growing scientific interest in ammonium nitrate thermal stability reflects increasing concerns regarding storage safety, thermal decomposition, explosion hazards, and performance optimization of ammonium nitrate-based materials. Despite the expanding body of literature, a comprehensive understanding of the intellectual structure, research evolution, and emerging trends within this field remains limited. Therefore, this study presents a bibliometric review aimed at mapping the knowledge structure and scientific development of research related to ammonium nitrate thermal stability. Bibliographic data were retrieved from the Scopus database using the search query TITLE-ABS-KEY («ammonium nitrate») AND TITLE-ABS-KEY («thermal stability»). A total of 247 publications were analyzed using Bibliometrix, Biblioshiny, and VOSviewer. Various bibliometric techniques, including citation analysis, co-occurrence analysis, co-authorship analysis, thematic mapping, Multiple Correspondence Analysis (MCA), Bradford’s Law, Lotka’s Law, and keyword evolution analysis, were employed to evaluate the development and conceptual organization of the research field. The conceptual structure analysis highlighted the multidisciplinary nature of the field, integrating knowledge from chemistry, materials science,

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chemical engineering, environmental science, and industrial safety. Furthermore, the findings revealed emerging opportunities associated with mineral-based stabilization technologies. Natural aluminosilicate materials such as bentonite and glauconite were identified as promising yet underexplored additives with potential applications in thermal stabilization, caking reduction, and safety enhancement of ammonium nitrate products.

Аннотация

Растущий научный интерес к термической стабильности аммиачной селитры обусловлен возрастающим вниманием к вопросам безопасности её хранения, термического разложения, предотвращения взрывоопасных ситуаций, а также оптимизации эксплуатационных характеристик материалов на основе аммиачной селитры. Несмотря на значительный рост числа научных публикаций, целостное представление об интеллектуальной структуре, эволюции исследований и современных тенденциях развития данного научного направления до настоящего времени остается недостаточно изученным. В связи с этим настоящее исследование представляет собой библиометрический обзор, направленный на картирование структуры знаний и научной эволюции исследований, посвящённых термической стабильности аммиачной селитры. Библиографические данные были получены из базы данных Scopus с использованием поискового запроса TITLE-ABS-KEY («ammonium nitrate») AND TITLE-ABS-KEY («thermal stability»). В общей сложности было проанализировано 247 научных публикаций с применением программных пакетов Bibliometrix, Biblioshiny и VOSviewer. Для оценки динамики развития, концептуальной организации и интеллектуальной структуры исследуемой области использовались различные библиометрические методы, включая анализ цитирования, анализ совместной встречаемости ключевых слов, анализ соавторства, тематическое картирование, анализ множественных соответствий (Multiple Correspondence Analysis, MCA), закон Брэдфорда (Bradford’s Law), закон Лотки (Lotka’s Law), а также анализ эволюции ключевых слов. Результаты анализа концептуальной структуры продемонстрировали междисциплинарный характер рассматриваемой области исследований, объединяющей достижения химии, материаловедения, химической технологии, экологических наук и промышленной безопасности. Кроме того, полученные результаты свидетельствуют о формировании новых перспективных направлений исследований, связанных с применением технологий стабилизации на основе минеральных материалов. В частности, природные алюмосиликатные материалы, такие как бентонит и глауконит, были идентифицированы как перспективные, однако недостаточно изученные минеральные добавки, обладающие высоким потенциалом для повышения термической стабильности аммиачной селитры, снижения её слёживаемости и улучшения эксплуатационной безопасности соответствующих продуктов.

Keywords: ammonium nitrate, thermal stability, bibliometric analysis, science mapping, knowledge structure, VOSviewer, Bibliometrix, thermal decomposition, thermodynamic stability, decomposition kinetics, keyword evolution, Multiple Correspondence Analysis, Bradford’s Law, Lotka’s Law, polymer modification, mineral additives, bentonite, glauconite.

Ключевые слова: аммиачная селитра, термическая стабильность, библиометрический анализ, картирование науки, структура знаний, VOSviewer, Bibliometrix, термическое разложение, термодинамическая стабильность, кинетика разложения, эволюция ключевых слов, анализ множественных соответствий (MCA), закон Брэдфорда, закон Лотки, полимерная модификация, минеральные добавки, бентонит, глауконит.

Introduction

Ammonium nitrate (AN) is one of the most extensively utilized inorganic compounds in modern agriculture and industry. Due to its high nitrogen content, favorable agronomic performance, and relatively low production cost, ammonium nitrate has become an essential component of nitrogen-based fertilizers worldwide. The growing demand for agricultural productivity and food security has contributed significantly to the continued use of ammonium nitrate in fertilizer manufacturing. As a result, considerable scientific attention has been devoted to improving its physicochemical properties, storage characteristics, and operational safety. In addition to its agricultural importance, ammonium nitrate plays a critical role in the production of energetic materials. Its strong oxidizing capability makes it suitable for various industrial applications, including blasting agents, mining explosives, and propellant formulations. The dual significance of ammonium nitrate in both agricultural and industrial sectors has stimulated extensive research aimed at understanding its behavior under different environmental and operating conditions.

Over the past several decades, advancements in thermal analysis techniques, materials science, and chemical engineering have enabled researchers to investigate the fundamental properties of ammonium nitrate in greater detail. These efforts have contributed to a deeper understanding of its decomposition mechanisms, thermal behavior, phase transformations, and interactions with various additives and modifiers. Consequently, ammonium nitrate has remained an important subject of scientific investigation across multiple disciplines, including chemistry, chemical engineering, materials science, environmental science, and industrial safety.

Despite its widespread application, ammonium nitrate presents several challenges associated with thermal stability and safe handling. The compound undergoes a series of temperature-dependent phase transitions and decomposition reactions that can significantly influence its physical properties and performance. Under certain conditions, thermal decomposition may generate large quantities of gaseous products, resulting in pressure buildup and increased safety risks. The thermal behavior of ammonium nitrate is influenced by numerous factors, including temperature, moisture content, impurities, particle size distribution, storage conditions, and the presence of catalytic substances. Variations in these parameters may alter decomposition pathways and affect the overall stability of the material. Consequently, understanding the mechanisms governing thermal degradation has become a major research priority.

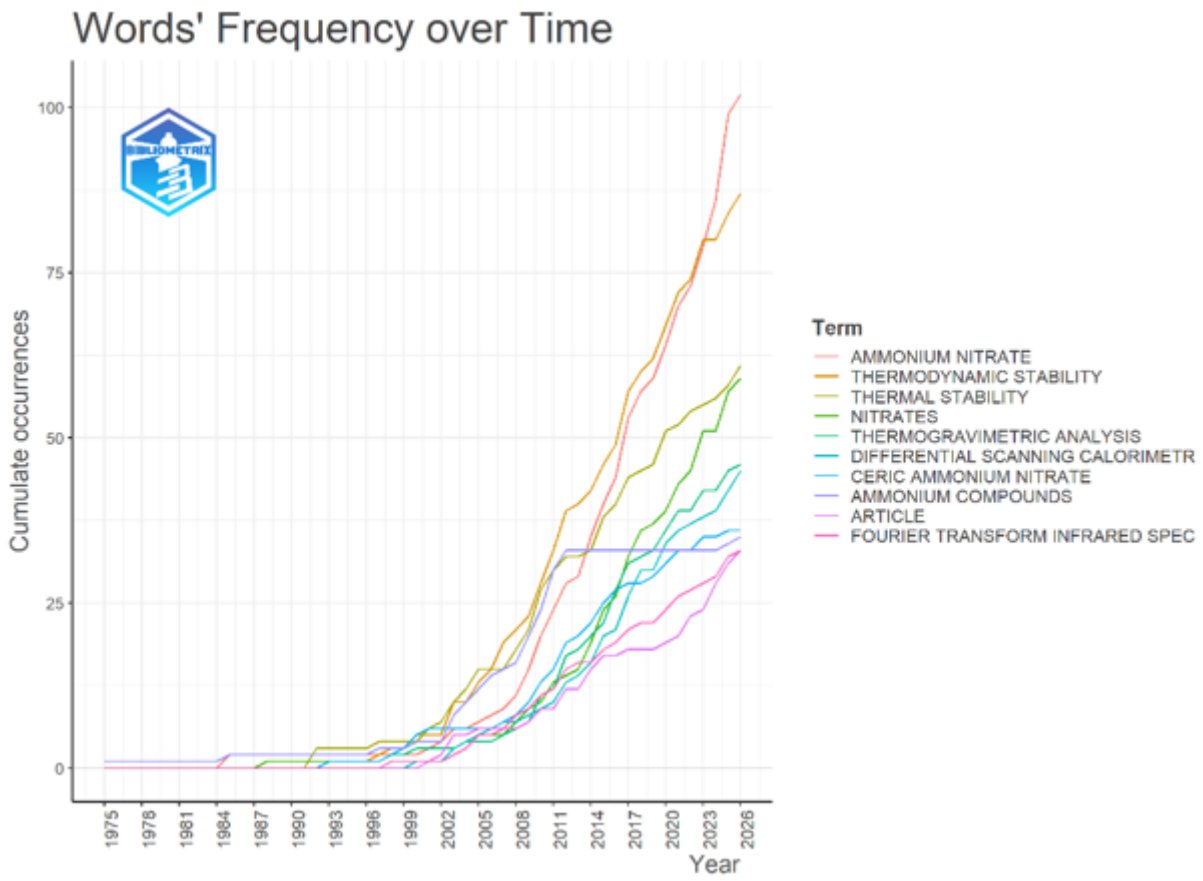

Figure 1. Temporal Evolution of the Most Frequent Keywords in Ammonium Nitrate Thermal Stability Research

Fig. 1 presents the cumulative occurrence of the most frequently used keywords throughout the study period.

The analysis reveals a substantial increase in the usage of terms such as «ammonium nitrate», «thermodynamic stability», and «thermal stability», indicating growing scientific interest in thermal behavior, decomposition mechanisms, and safety-related aspects of ammonium nitrate systems.

Industrial incidents involving ammonium nitrate have further highlighted the importance of thermal stability studies. Accidental decomposition and explosion events have demonstrated the potential hazards associated with improper storage, contamination, and exposure to elevated temperatures.

These concerns have encouraged both researchers and industry practitioners to develop strategies aimed at enhancing thermal resistance, improving storage stability, and reducing safety risks throughout the product life cycle. In recent years, increasing attention has been directed toward the development of stabilization technologies based on polymers, mineral additives, surface coatings, and composite materials. Such approaches seek to improve the thermal performance of ammonium nitrate while maintaining its functionality and economic viability for large-scale industrial applications.

The rapid expansion of scientific literature related to ammonium nitrate thermal stability has created a need for systematic methods capable of evaluating research progress and identifying emerging trends. Traditional review articles provide valuable qualitative insights; however, they

often face limitations when attempting to synthesize large volumes of publications produced over extended periods. Bibliometric analysis offers a quantitative approach for examining the structure, development, and dynamics of scientific research.

By analyzing publication records, citation patterns, collaboration networks, and keyword relationships, bibliometric methods enable researchers to identify influential contributors, highly cited studies, dominant research themes, and evolving scientific directions. Furthermore, science mapping techniques facilitate the visualization of conceptual relationships and knowledge structures within a research field.

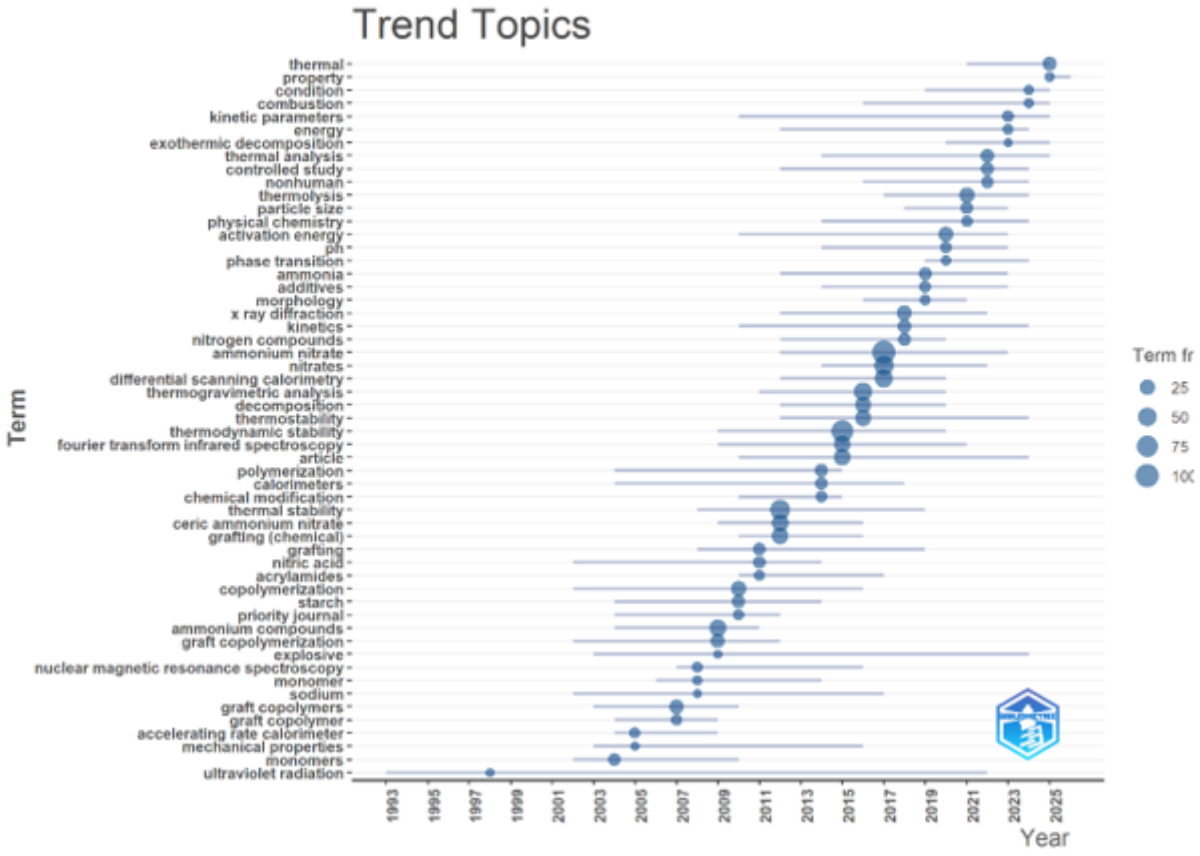

Figure 2. Trend Topics in Ammonium Nitrate Thermal Stability Research

Fig. 2 depicts the temporal evolution of research topics identified from author keywords. The figure demonstrates a transition from fundamental studies involving thermal decomposition and calorimetric analysis toward advanced themes such as kinetic modeling, polymer modification, thermodynamic behavior, and stabilization strategies.

Research Objectives

Given the growing body of literature on ammonium nitrate thermal stability, a comprehensive evaluation of research trends and knowledge structures is necessary to better understand the evolution of this field. Therefore, the present study aims to provide a systematic bibliometric overview of global research related to ammonium nitrate thermal stability. Specifically, this study seeks to analyze publication growth patterns, identify the most productive authors, institutions, countries, and journals, and evaluate the intellectual and conceptual structure of the field through citation, co-occurrence, and collaboration analyses. In addition, the study investigates thematic

evolution, research hotspots, and emerging scientific directions that have shaped the development of ammonium nitrate thermal stability research over time. By integrating bibliometric indicators with knowledge-mapping techniques, this work provides a comprehensive understanding of the current research landscape and identifies future opportunities associated with thermal stabilization technologies, advanced material modification strategies, and the development of safer ammonium nitrate-based products.

Materials and Methods

This study employed the Scopus database as the primary source of bibliographic information. Scopus was selected because of its extensive coverage of peer-reviewed scientific literature across multiple disciplines, including chemistry, chemical engineering, materials science, environmental science, and industrial safety. The database provides comprehensive bibliographic records, citation information, author affiliations, and keyword metadata, making it particularly suitable for bibliometric and scientometric investigations. All bibliographic records were retrieved directly from Scopus to ensure consistency in data collection and avoid discrepancies that may arise when combining multiple databases. The retrieved records constituted the foundation for subsequent analyses of publication trends, scientific impact, collaboration networks, conceptual structures, and thematic evolution within the field of ammonium nitrate thermal stability research. A systematic search strategy was developed to identify publications specifically related to the thermal stability of ammonium nitrate. The search was conducted within the titles, abstracts, and author keywords indexed in the Scopus database using the following search query: TITLE-ABS-KEY («ammonium nitrate») AND TITLE-ABS-KEY («thermal stability»).

Fig. 3 presents the local impact of the most influential journals measured by the H-index within the analyzed dataset. The Journal of Applied Polymer Science, Journal of Thermal Analysis and Calorimetry, and Thermochimica Acta emerged as the leading publication sources, reflecting their significant contribution to the development of ammonium nitrate thermal stability research.

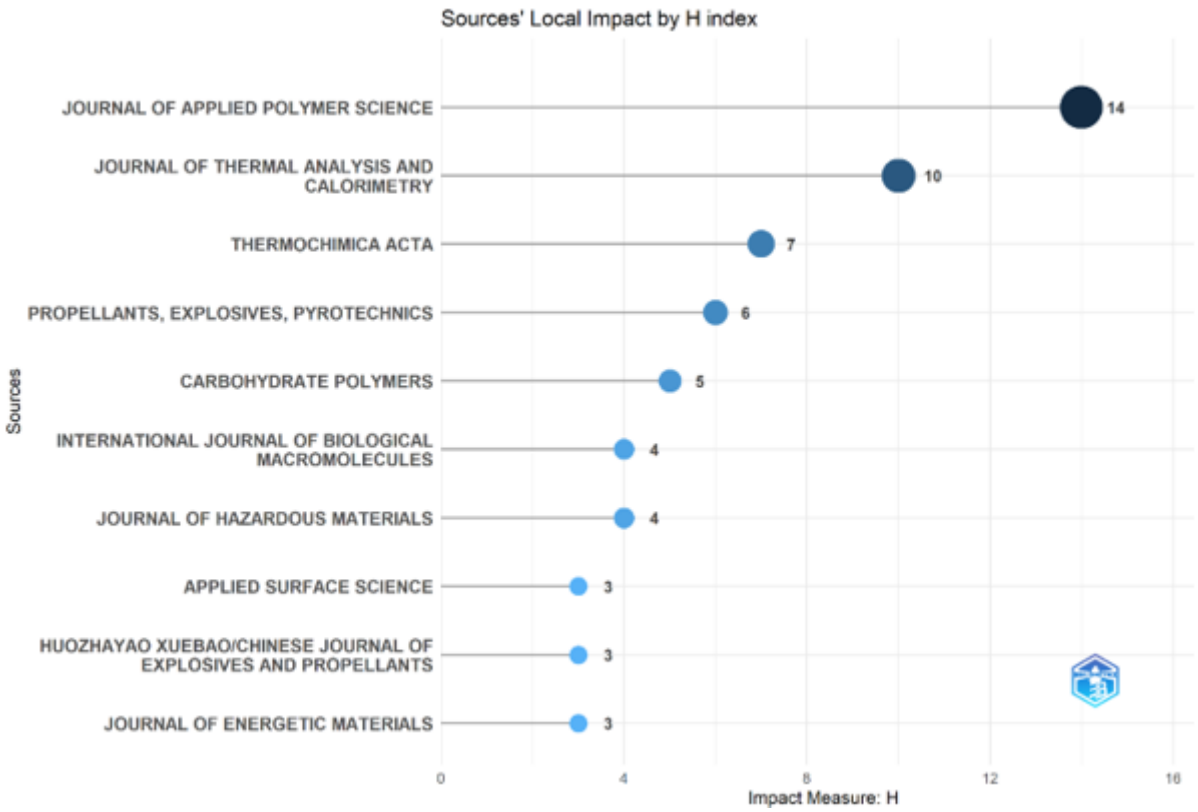

Figure 3. Sources' Local Impact Based on H-Index

Figure 4. Most Relevant Keywords in the Research Field

Fig. 4 depicts the most frequently occurring keywords extracted from the analyzed publications. «Ammonium nitrate» represents the dominant research theme, followed by «thermodynamic stability», «thermal stability», «thermogravimetric analysis», and «differential scanning calorimetry», highlighting the central role of thermal characterization techniques in this field. The search was performed on a single date to ensure consistency of the dataset and to minimize variations associated with database updates. All available records matching the search criteria were exported in CSV format. The exported data included information on publication titles, authors, affiliations, abstracts, author keywords, source titles, publication years, references, and citation counts. The initial search yielded 247 publications. These records were subsequently examined and prepared for bibliometric analysis.

The final dataset represents the global scientific literature associated with ammonium nitrate thermal stability and reflects contributions from a wide range of scientific disciplines and research communities. To ensure the reliability and relevance of the analysis, specific inclusion and exclusion criteria were applied during the dataset preparation process. The inclusion criteria comprised publications that explicitly addressed topics related to ammonium nitrate thermal stability, thermal decomposition, thermodynamic behavior, calorimetric analysis, safety assessment, stabilization technologies, or related physicochemical properties.

Only documents indexed in the Scopus database and containing the selected search terms within their titles, abstracts, or keywords were considered eligible for inclusion.

Publications unrelated to ammonium nitrate thermal behavior were excluded from the analysis. Duplicate records, incomplete entries, and documents lacking essential bibliographic information were also removed where necessary. The screening process was conducted to ensure that the final dataset accurately reflected the research domain under investigation and minimized the influence of irrelevant publications.

Fig. 5 presents the most productive research institutions contributing to studies on ammonium nitrate thermal stability.

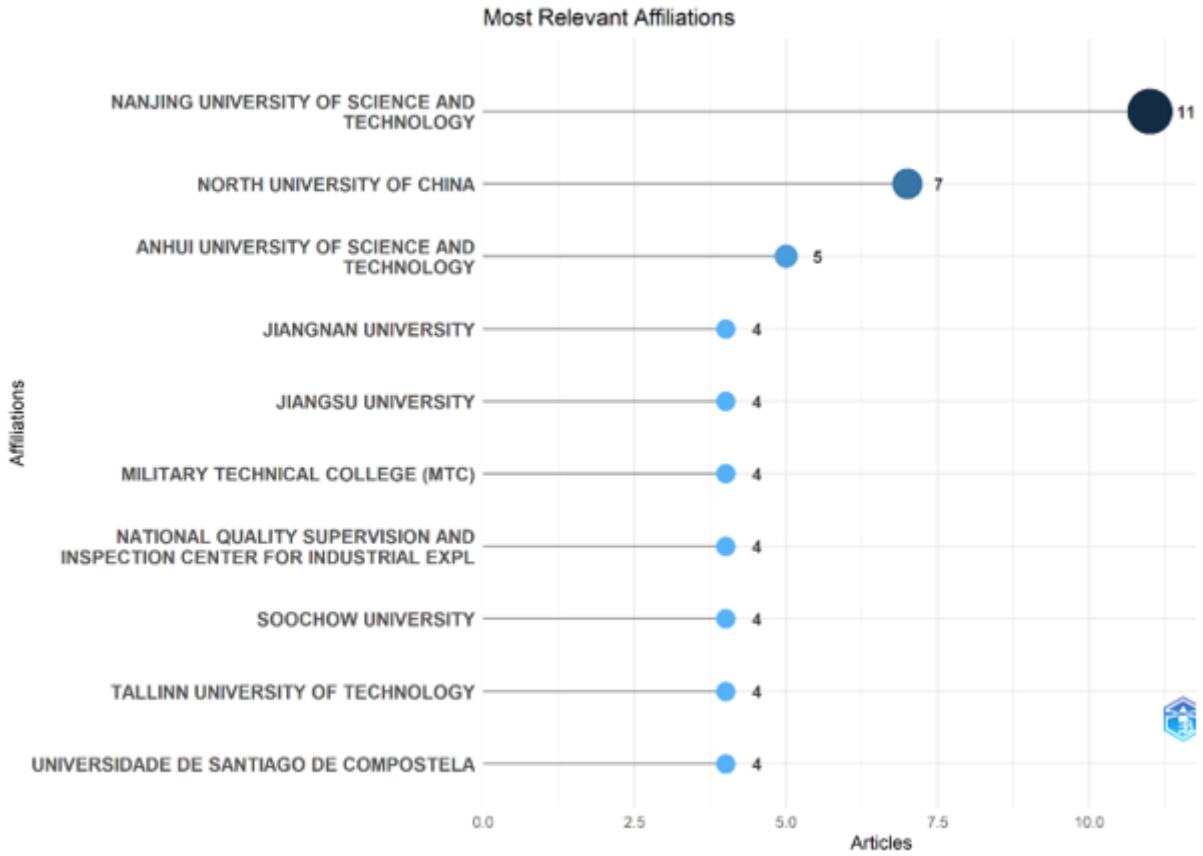

Figure 5. Most Relevant Research Affiliations

The results indicate that Chinese universities and research centers dominate publication output, demonstrating their leading role in advancing thermal analysis, energetic materials, and stabilization technologies.

Fig. 6 depicts the most globally cited publications identified within the dataset. These highly influential studies have substantially shaped current understanding of ammonium nitrate decomposition mechanisms, thermal behavior, environmental impacts, and material modification approaches. Prior to analysis, the retrieved bibliographic data underwent a preprocessing procedure to improve data quality and analytical accuracy. The dataset was examined for duplicate entries, inconsistencies in author names, variations in institutional affiliations, and differences in keyword terminology.

Keyword standardization was performed to merge synonymous expressions and reduce fragmentation within the keyword network. Variations in spelling, abbreviations, and singular-plural forms were carefully reviewed to ensure consistency across the dataset.

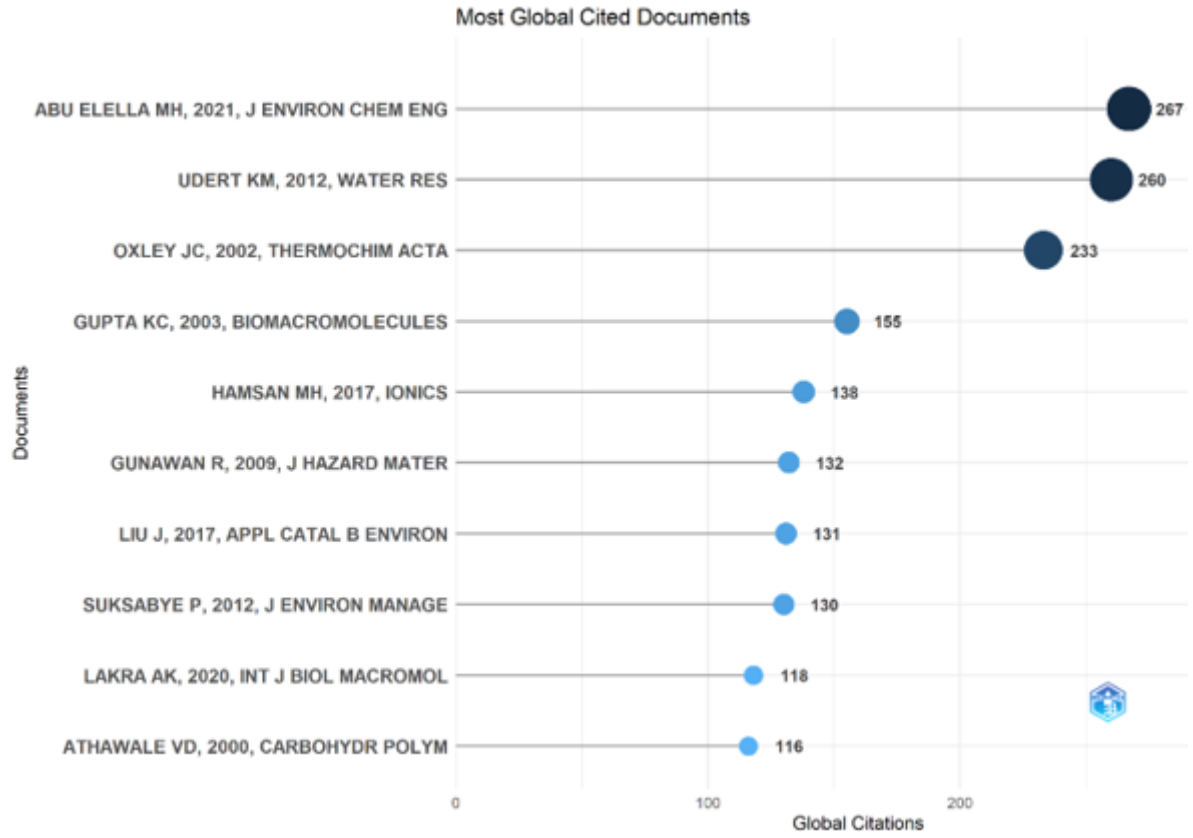

Figure 6. Most Globally Cited Documents in Ammonium Nitrate Thermal Stability Research

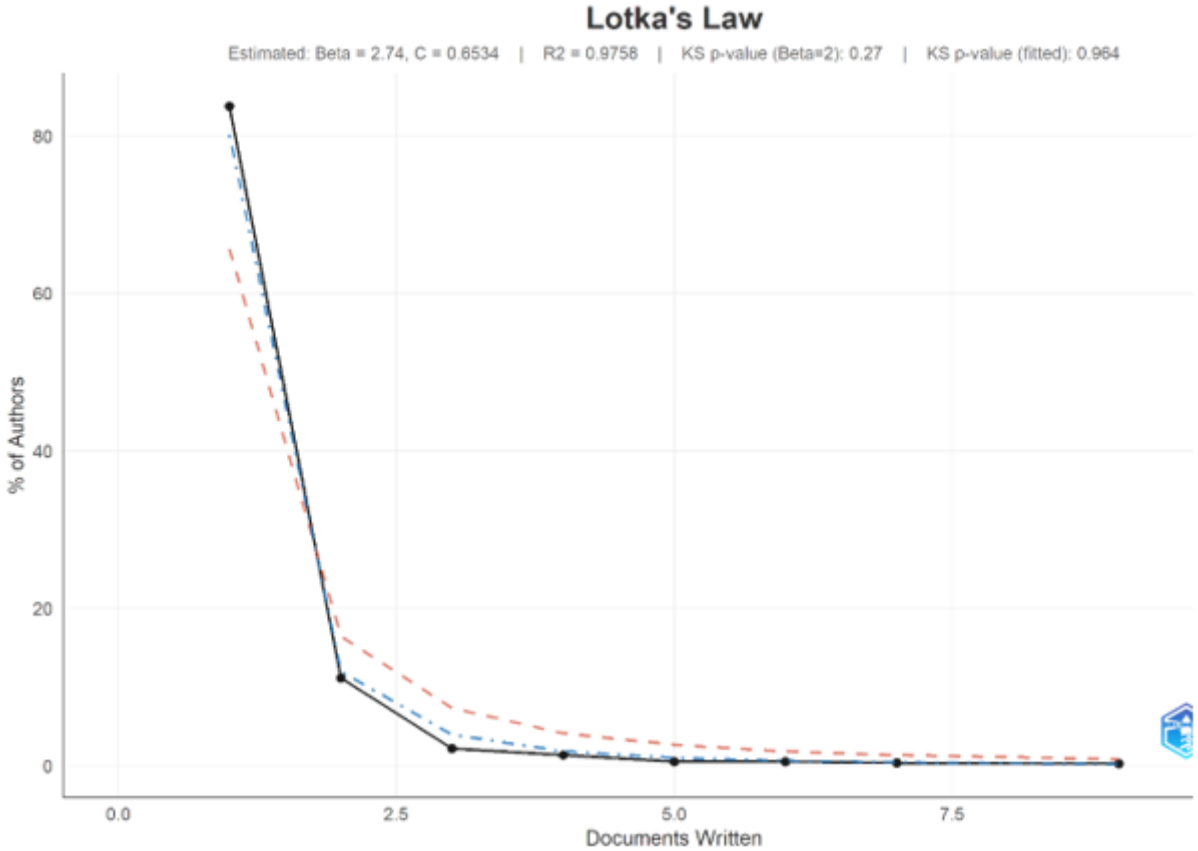

Figure 7. Author Productivity Distribution According to Lotka's Law

Author names and institutional affiliations were also checked for possible inconsistencies that could affect productivity and collaboration analyses.The cleaned dataset was subsequently converted into formats compatible with the selected bibliometric software packages. This preprocessing stage enhanced the reliability of network visualizations and improved the interpretation of thematic relationships among publications.

Fig. 7 presents the distribution of author productivity based on Lotka's Law. The results indicate that the majority of authors contributed only a small number of publications, whereas a limited group of highly productive researchers generated a substantial proportion of the scientific output in this field.

Bibliometric Analysis Tools

The bibliometric analyses were conducted using Bibliometrix, Biblioshiny, and VOSviewer, which are among the most widely used tools for science mapping and bibliometric investigations.

Bibliometrix, an open-source package developed within the R environment, was employed to calculate descriptive bibliometric indicators and evaluate publication performance.

Biblioshiny, the web-based interface of Bibliometrix, facilitated data exploration and visualization through an interactive analytical environment.

These tools were used to generate publication trends, source analyses, author productivity assessments, citation indicators, thematic maps, trend topic analyses, Bradford’s law distributions, and Lotka’s law evaluations.

Fig. 8 depicts a treemap representation of keyword frequency within the analyzed literature. Larger blocks correspond to more frequently occurring terms, illustrating the predominance of topics related to ammonium nitrate, thermal stability, thermodynamic analysis, decomposition behavior, and polymer-modified systems.

Conclusions

This study presents a comprehensive bibliometric assessment of the global scientific literature on ammonium nitrate thermal stability.

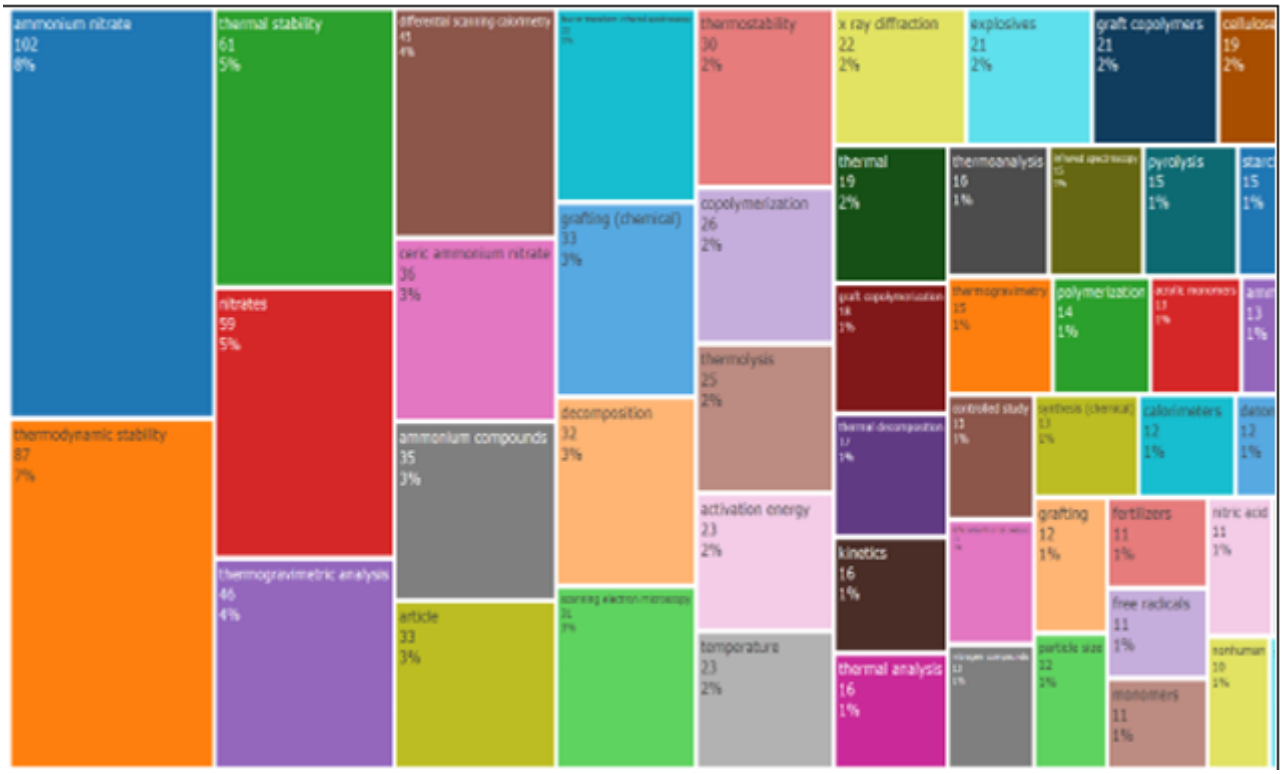

Figure 8. Treemap Visualization of the Most Frequent Research Keywords

By integrating performance analysis with science mapping techniques, the study provides a detailed overview of the development, intellectual structure, and emerging directions of research within this field.

Fig. 9 presents the conceptual structure of the research field generated using multiple correspondence analysis. The map identifies several thematic clusters associated with thermal analysis, decomposition kinetics, spectroscopic characterization, polymerization processes, and stabilization mechanisms, demonstrating the multidisciplinary nature of AN thermal stability research.

Fig. 10 depicts the geographical distribution of scientific production related to ammonium nitrate thermal stability. Countries with darker coloration exhibit higher publication output, highlighting China as the leading contributor, followed by several countries actively engaged in thermal analysis, energetic materials, and fertilizer safety research. The findings reveal a steady expansion of scientific activity related to ammonium nitrate thermal stability, particularly during the last decade. The increasing number of publications reflects growing academic and industrial interest in understanding the thermal behavior, decomposition mechanisms, safety characteristics, and stabilization strategies associated with AN-based materials.

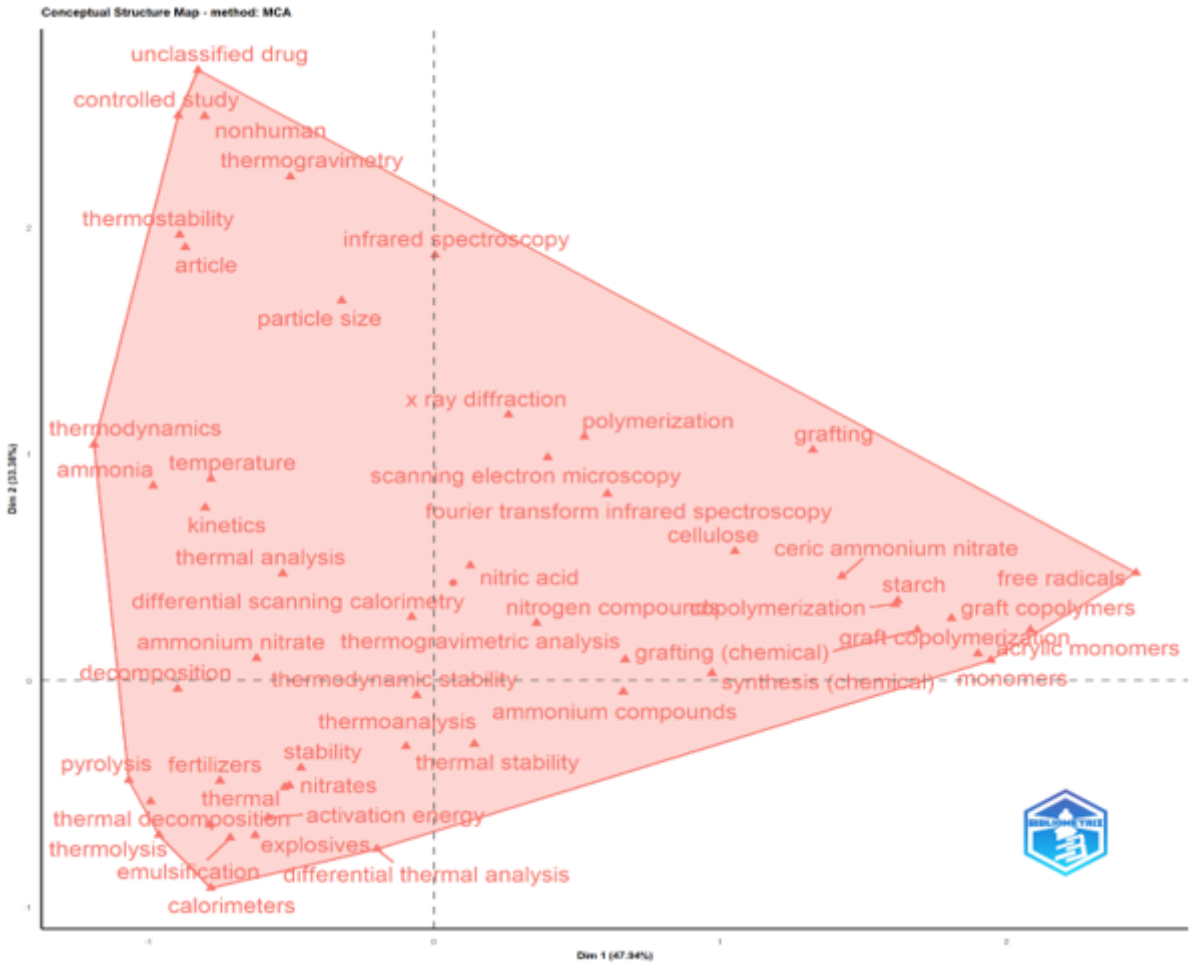

Figure 9. Conceptual Structure Map Based on Multiple Correspondence Analysis (MCA)

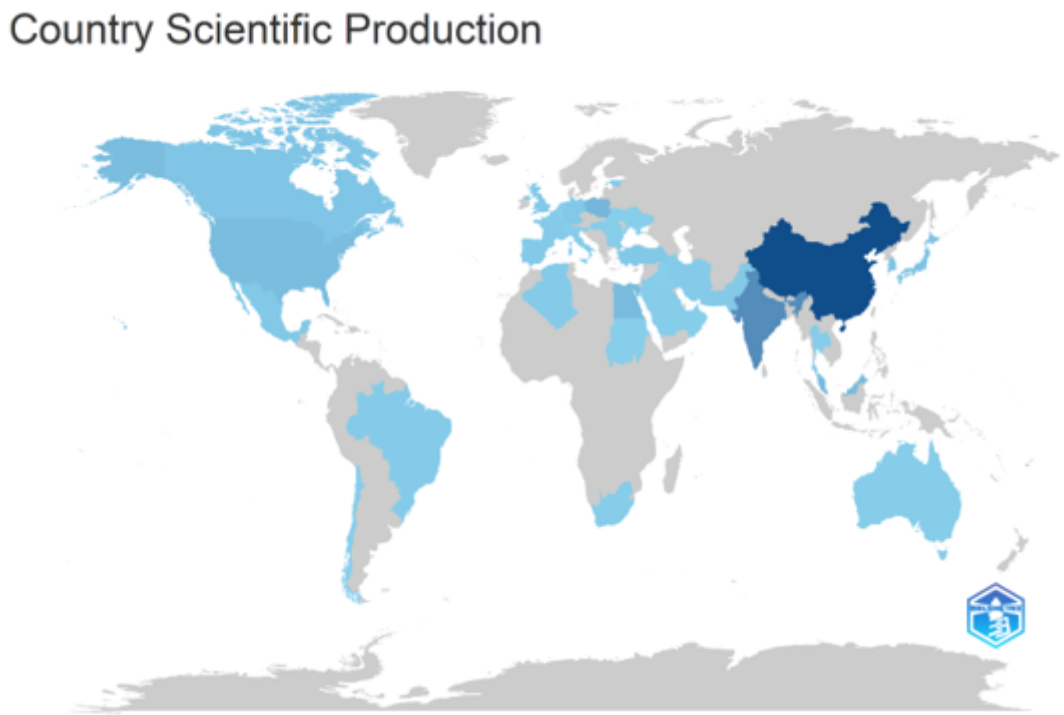

Figure 10. Global Distribution of Scientific Production by Country

This trend demonstrates the continued relevance of thermal stability research in addressing both technological and safety-related challenges. The analysis identified significant contributions from a relatively limited number of countries, institutions, and researchers. China emerged as the leading contributor in terms of scientific productivity and research impact, supported by strong institutional participation and extensive publication activity. The collaboration networks further indicate the existence of active international partnerships that contribute to knowledge exchange and scientific advancement in this research domain.

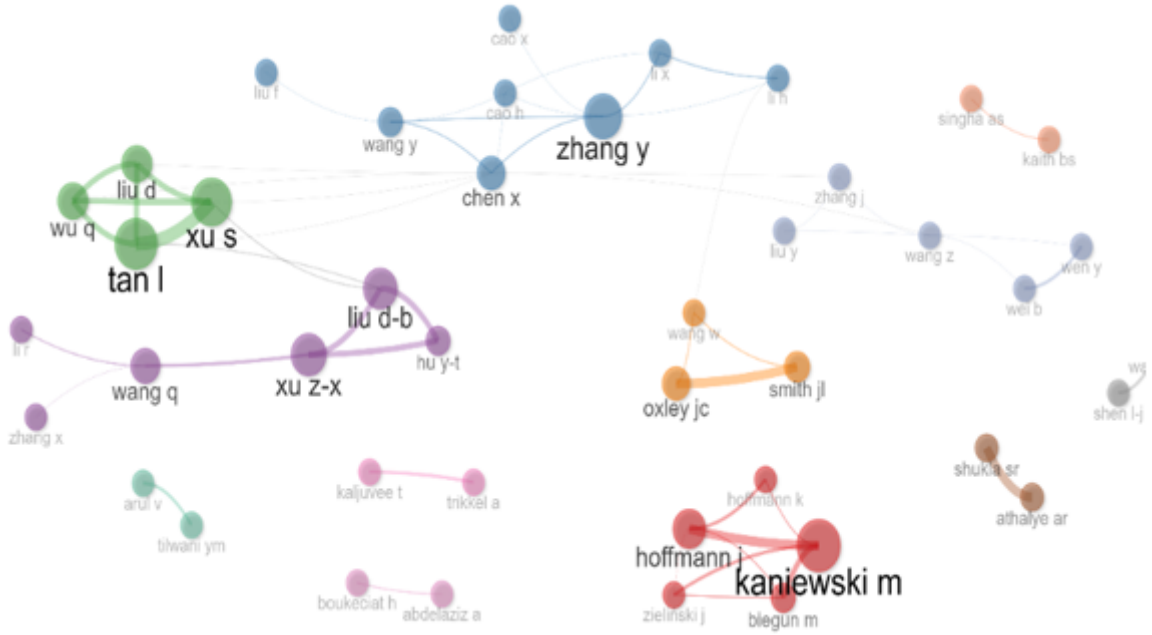

Figure 11. Author Collaboration Network

Fig. 11 presents the co-authorship network of the most productive researchers. Distinct collaboration clusters are observed, indicating active scientific partnerships and knowledge exchange among leading authors working on thermal decomposition, stability enhancement, and characterization of ammonium nitrate systems.

Fig. 12 depicts the distribution of scientific articles according to Bradford's Law. The analysis identifies a core group of highly productive journals responsible for publishing a significant proportion of the literature, followed by broader zones containing journals with progressively lower publication frequencies.

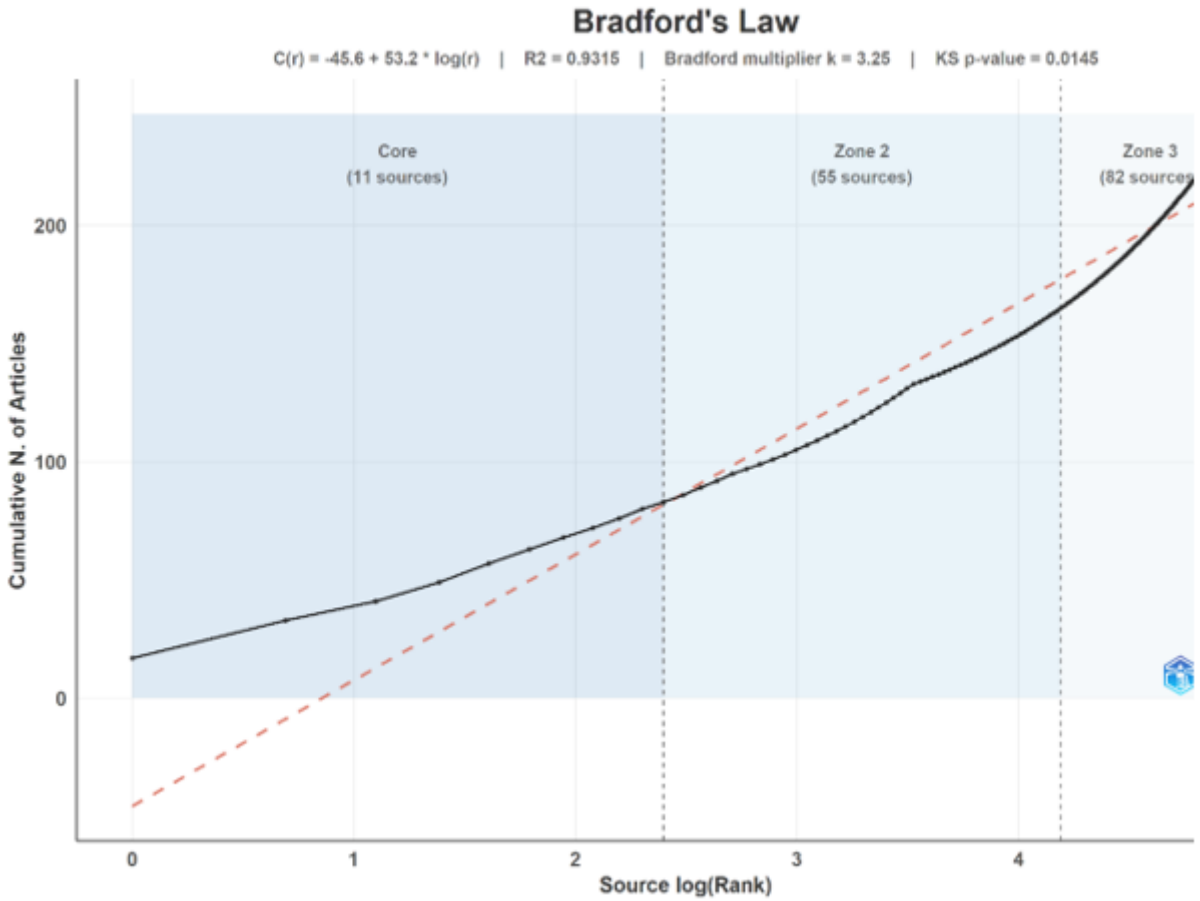

Figure 12. Bradford's Law Analysis of Source Distribution

Keyword co-occurrence, thematic mapping, and conceptual structure analyses demonstrated that thermal decomposition, thermodynamic stability, thermal analysis, and decomposition kinetics remain the principal research themes.

These topics form the scientific foundation of the field and continue to drive investigations aimed at improving the understanding of ammonium nitrate behavior under various thermal conditions.

The intellectual structure of the literature further highlights the multidisciplinary nature of the field, incorporating concepts from chemistry, materials science, chemical engineering, environmental studies, and industrial safety.

Fig. 13 presents the local scientific impact of the most influential authors measured by the H-index. The identified researchers have contributed substantially to the advancement of knowledge regarding ammonium nitrate thermal stability, decomposition kinetics, and safety enhancement technologies.

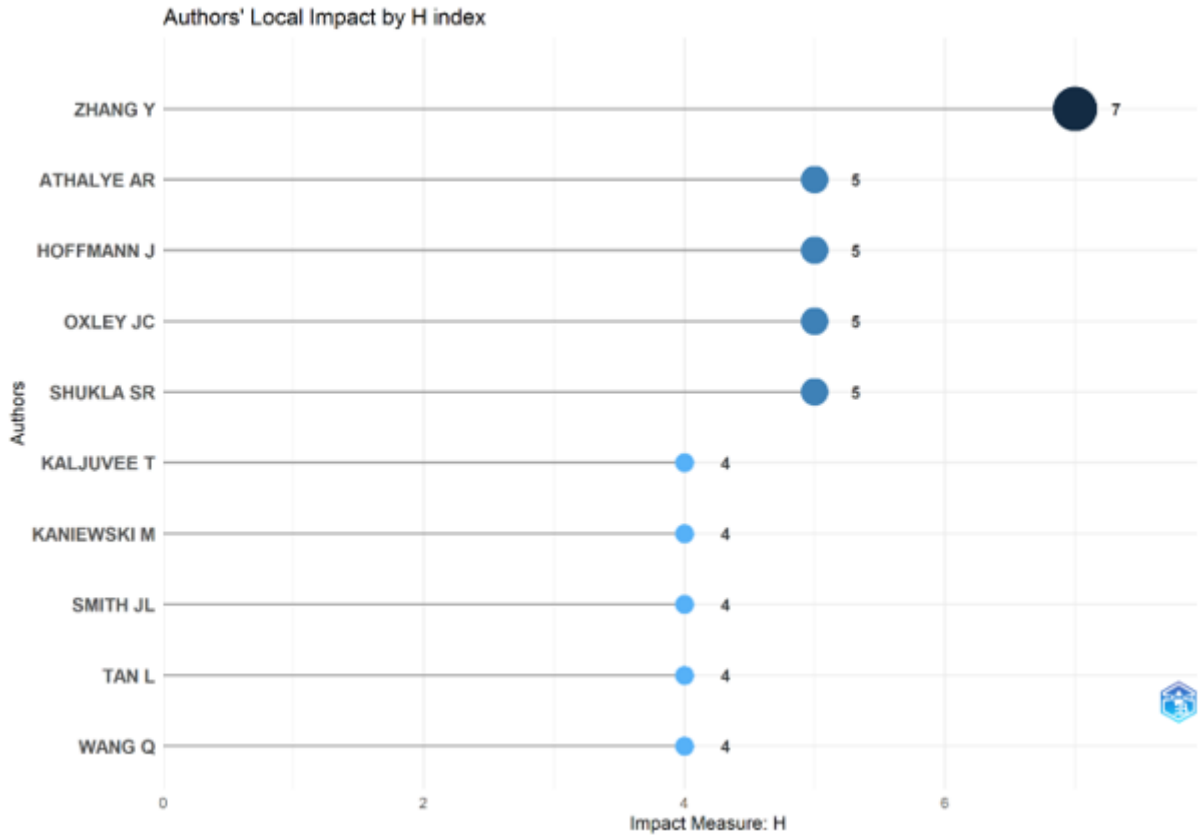

Figure 13. Authors' Local Impact Based on H-Index

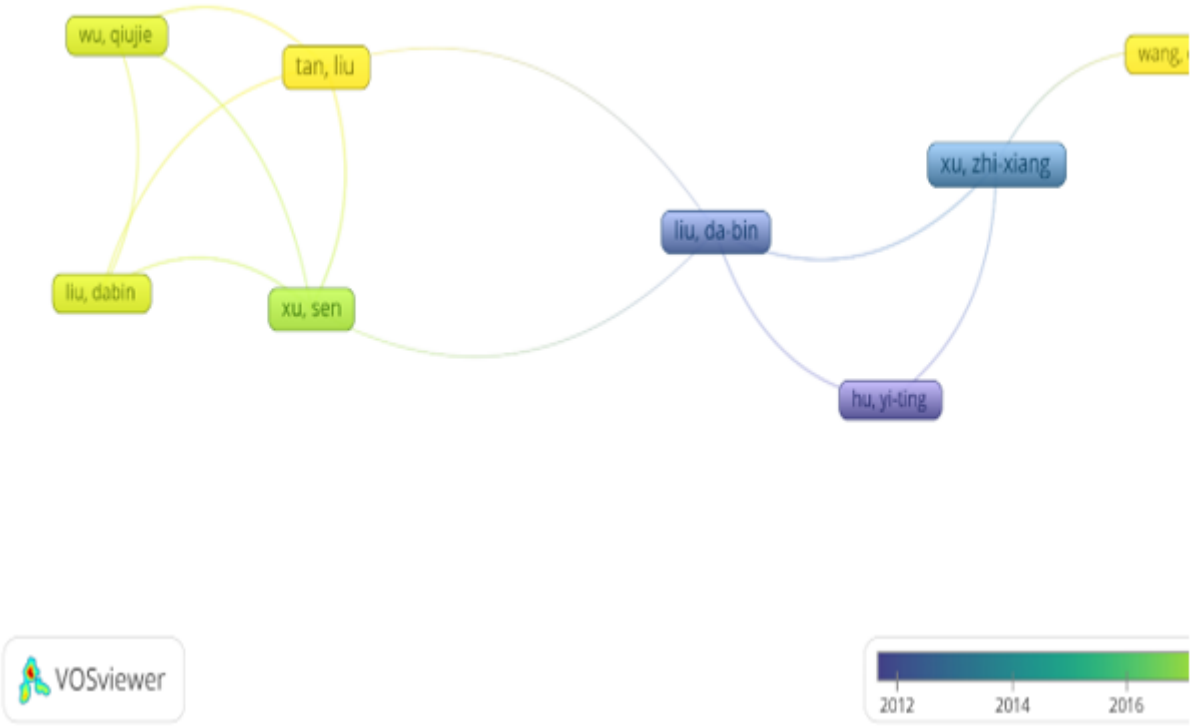

Figure 14. Keyword Evolution Network

Fig. 14 depicts the temporal evolution of research keywords across different time periods. The network illustrates how research themes have evolved from fundamental investigations of ammonium nitrate and thermal stability toward advanced studies involving copolymerization, graft

polymers, and material modification technologies. The evolution of research themes revealed a gradual transition from fundamental thermal characterization studies toward application-oriented approaches involving material modification and performance enhancement.

In particular, increasing attention has been directed toward polymer-based stabilization technologies, advanced characterization methods, and strategies designed to improve thermal resistance and operational safety. The emergence of these themes reflects the growing emphasis on developing practical solutions to challenges associated with storage, transportation, and industrial utilization of ammonium nitrate.

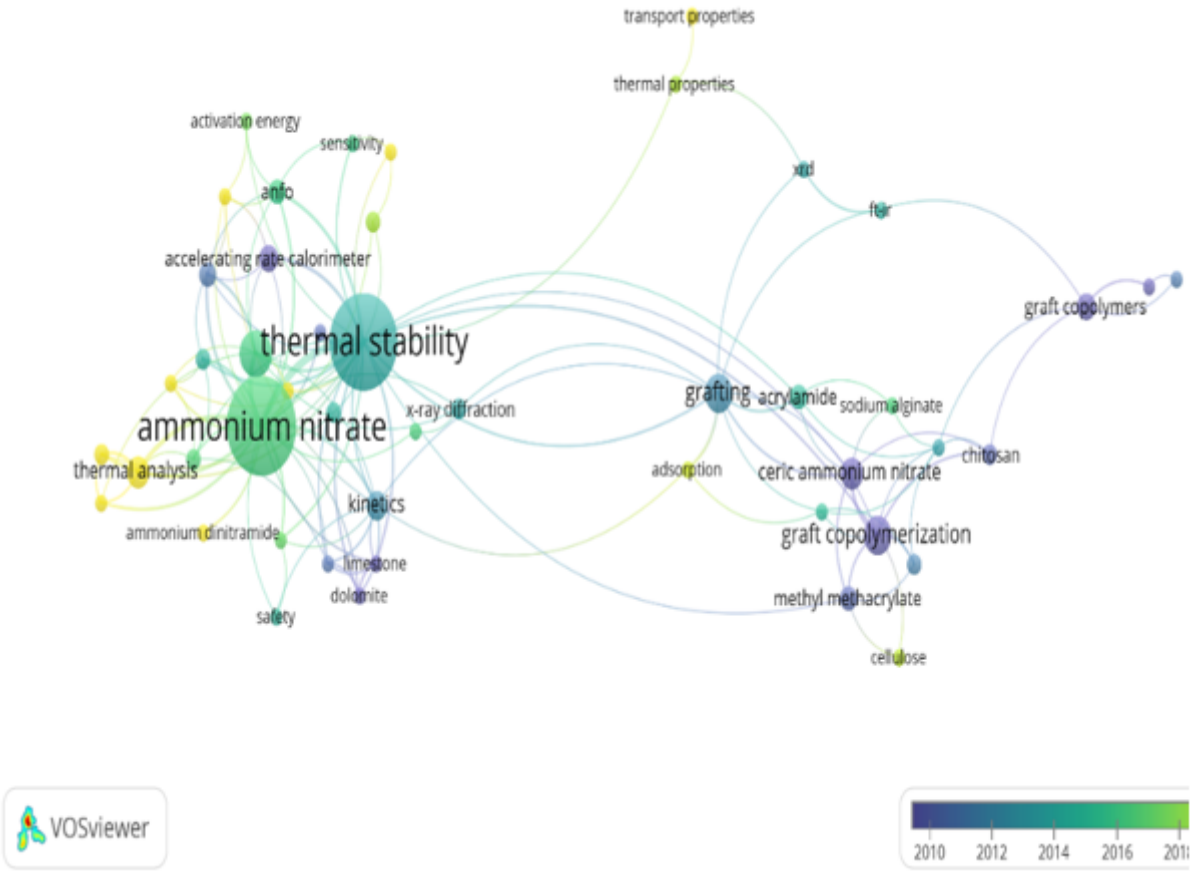

Figure 15. Overlay Visualization of Keyword Co-Occurrence Network

Fig. 15 presents an overlay visualization of the keyword co-occurrence network generated using VOSviewer. The color gradient reflects the temporal evolution of research topics, with recent studies increasingly focusing on polymer-based modification strategies, advanced stabilization approaches, and thermal performance optimization of ammonium nitrate materials.

The results also suggest that several promising research opportunities remain insufficiently explored. Among these, mineral-based stabilization approaches represent a particularly important direction for future investigation. Natural aluminosilicate materials, including bentonite and glauconite, possess physicochemical properties that may contribute to improved thermal stability, reduced caking behavior, and enhanced storage safety. Despite their potential advantages, these materials remain underrepresented within the existing literature and warrant more comprehensive experimental and mechanistic studies.

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Current state of polymer binders used in particleboard production: a review

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Современное состояние полимерных связующих материалов, используемых в производстве ДСП: обзор

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Abstract

The article presents the results of research on the influence of reactive compounds on the hardening process of both modified and unmodified carbamide-formaldehyde resin derivatives, and determines the optimal hardening regimes for their application in the production of wood-plastic tile materials.

Increasing requirements for the strength, performance, and durability of wood-plastic boards necessitate the modification of carbamide-formaldehyde resins using physical and chemical methods. This modification is aimed at improving the adhesive, physical-mechanical, and technological properties of the binder.

As a result of studying the physicochemical properties of unmodified and modified resins, it was established that an increase in the content of modifying additives is accompanied by an increase in the concentration of chlorine ions in the resin. In this case, the hardening time changes in the following sequence: polyvinyl chloride → epichlorohydrin → benzyl chloride. It has been established that introducing a modifier in an amount exceeding 10 % of the resin mass is inappropriate, as it leads to a significant increase in its hardening time and a deterioration of the process's technological indicators.

Аннотация

В статье представлены результаты исследований влияния реактивных соединений на процесс отверждения как модифицированных, так и немодифицированных производных карбамидоформальдегидной смолы, а также определены оптимальные режимы отверждения для их применения в производстве древесно-пластиковых плиток.

Повышение требований к прочности, эксплуатационным характеристикам и долговечности древесно-пластиковых плит обуславливает необходимость модификации карбамидоформальдегидных смол физико-химическими методами. Эта модификация направлена на улучшение адгезионных, физико-механических и технологических свойств связующего.

В результате изучения физико-химических свойств немодифицированных и модифицированных смол установлено, что увеличение содержания модифицирующих добавок сопровождается увеличением концентрации хлорид-ионов в смоле. При этом время отверждения изменяется в следующей последовательности: поливинилхлорид → эпихлоргидрин → бензилхлорид. Установлено, что введение модификатора в количестве, превышающем 10 % от массы смолы, нецелесообразно, поскольку это приводит к значительному увеличению времени ее отверждения и ухудшению технологических показателей процесса.

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Keywords: polymer, urea-formaldehyde resin, modification, reactive compounds, benzene chloride, epichlorohydrin, polyvinyl chloride, gossypol resin, lignin, composite wood-plastic plate material.

Ключевые слова: полимер, карбамидоформальдегидная смола, модификация, реакционноспособные соединения, хлористый бензол, эпихлоргидрин, поливинилхлорид, госсиполовая смола, лигнин, композиционный древесно-пластиковый плитный материал.

Introduction

Wood-plastic materials are among the most dynamically developing types of woodworking industry products and hold one of the leading positions in the world in terms of production growth rates. Their widespread use is due to a number of valuable properties, including high uniformity of structure and characteristics across the entire volume of the material, size stability under changing humidity conditions, as well as good operational performance.

An additional advantage is the technological ease of manufacturing products of various shapes and configurations, including large-format sheet materials. The possibility of using affordable polymer binders and using single-year plant stems as raw materials expands the raw material base of production and contributes to the wider implementation of wood-plastic materials in various industrial sectors. [3; 7; 10; 13].

Research object. To study and analyze the states of polymer binders used in the production of particleboard (PB) and wood-plastic materials and boards (WPMB), we examined phenol-formaldehyde, urea-formaldehyde, and other polymer resins in this article.

Study results and their analysis. Let us consider the phenol-formaldehyde and urea-formaldehyde resins widely used in the production of PB and WPMB [4; 8; 9].

Phenol-formaldehyde resins are the primary component of adhesive compositions, possessing a complex of valuable properties and found wide application in various industrial sectors. Establishing the relationship between composition, structure, and properties is one of the key tasks of the modern stage in the development of adhesive material production technologies. Solving this task will allow for the purposeful development of composite materials and adhesive compositions with a specified set of properties, including increased heat resistance and weather resistance, as well as significantly simplify and intensify the technological process and improve the quality of finished products. [2].

These resins are used as a binding component in the production of filled press compositions with various fillers (cellulose, glass fiber, wood flour), wood-fiber and wood-wool boards, adhesives, and impregnating and filling compositions (for plywood, woven, and fiber-filled materials).

Phenol-formaldehyde resin is a substance of synthetic origin and is used for the manufacture of wood shavings. Phenol-formaldehyde resin ensures high stability and strength of adhesive compounds under the influence of hot and warm water, which is why it is classified as a highly water-resistant resin [1].

Phenol-formaldehyde resin is most widely used in the manufacture and bonding of WSB. Such resin hardens quite quickly and possesses very high adhesive strength and a light color. When gluing wood shavings, low-toxic SFJ-3014 grade resin is used, which complies with the accepted standard (GOST 20907-75*).

Table 1.

Physicochemical properties of SFJ-3014 phenol-formaldehyde resin

Name	Indicators
Content of non-volatile substances (dry residue), %	46-52
Viscosity according to VZ-4, s	17-90
Alkaline content, %	6.5 – 7.5
Free phenol content, %, not more than	0.10
Free formaldehyde content, %, not more than	0.15
After boiling in water for one hour, the strength limit of the plywood layer during cracking is -MPa, at least.	1.5

Phenol-formaldehyde resins are obtained by the polycondensation of phenol with formaldehyde. Phenol-formaldehyde resins of the resole type are formed at an equimolar reactant ratio or in the presence of excess formaldehyde under alkaline catalysis, whereas novolac resins are obtained in the presence of excess phenol under acidic conditions. The hardening process, i.e., the transformation into resite, occurs slowly at normal temperatures from 6 months to 1 year; at elevated temperatures, the hardening rate increases significantly. In the presence of acid catalysts, solution resins harden at a higher rate and at room temperature.

Resins in the resite stage are insoluble, insoluble, and possess fairly high heat resistance. At temperatures above 280 °C, they begin to decompose gradually. The study of the thermal destruction of phenol-formaldehyde resins showed that at these temperatures, the formation of diphenyl oxide bonds occurs, increasing the degree of system adhesion. During thermo-oxidative destruction, primarily, the oxidation of methylene groups to carboxyl groups occurs, which at a temperature of about 200 °C are capable of interacting to form heat-resistant polymers [11].

Novolac resin molecules do not contain methyl groups, and therefore are unable to undergo polycondensation reactions and do not form spatial structures. Novolac resins can be converted into an insoluble and non-melting state by treating them with formaldehyde, paraffin, and hexamethylene tetramine. Novolac resins are most often hardened using hexamethylene tetramine at elevated temperatures. Some characteristics of phenol-formaldehyde resins are shown in Table 2 [12].

Table 2.

Characteristics of phenol-formaldehyde resins

Name	Trade name	Molecular mass	Melting point, °C	Density, kg/m^3	Content of me thyl groups, %
p-third-Butylphenolformaldehyde resin	Phenofor B	500-600	65-80	1100	≥12
p-third-Octylphenol formaldehyde resin	Phenofor O	900-1200	75-90	1040	≥9
Bromethylated p-tert-butylphenolfor-mal-dehyde resin	Fenofor BB	1000-1400	60-80	-	≥10

To obtain adhesives, phenol-formaldehyde resins of the resolution type with a molecular weight of 700–1000 are primarily used. Novolactic phenol-formaldehyde resins are used significantly less frequently, primarily in modified adhesives. Resins from cresols and substituted phenols are of less interest for producing adhesives.

Phenol-formaldehyde resin is a transparent and homogeneous liquid in appearance, ranging from dark cherry-brown to red-brown, within the same color batch, without mechanical impurities. However, at a temperature of 100…105 °C, the hardening degree of SFJ-3014 grade resin is insufficient. At the same time, it possesses high alkalinity, which is associated with the conditions for synthesizing low-viscosity resins. Therefore, to manufacture slabs with increased water and atmospheric resistance suitable for use in elements of standard low-rise house construction, it is necessary to enhance the technological properties of the resin.

By modifying SFJ-3014 grade resin using aluminum sulfate, the deepening and acceleration of the hardening process are achieved, and water resistance is increased.

It should be noted that phenol-formaldehyde resin is not produced in our republic due to the lack of raw materials for obtaining phenol. Furthermore, it is very expensive and, accordingly, scarce; therefore, it will be necessary to purchase this product in foreign currency.

But the main disadvantage of phenol-formaldehyde resin is its toxicity.

Phenol-formaldehyde resins can have harmful effects on the skin, causing dermatitis and eczema [13]. Uncured phenol-formaldehyde resin can contain up to 11 % free phenol.

When phenol-formaldehyde resins are rejected in plastic (phenoplastics), the sewing of oligomeric resin fragments occurs with the participation of free phenol contained within it, while the content of phenol incorporated into the phenoplastic decreases to trace amounts; the sanitary regulations of the Russian Federation regulate the permissible quantities of phenol and formaldehyde migration for products made of phenoplastics; specifically, for products in contact with food products, for phenol – 0.05 mg/l, for formaldehyde – 0.1 mg/l.

Therefore, as noted above, it is necessary to modify the phenol-formaldehyde resin or replace it with another, less toxic resin, i.e., urea-formaldehyde (carbamide).

It should be noted that in our republic, urea-formaldehyde resin is primarily used as a polymer binder in the production of wood-plastic tile materials.

Urea-formaldehyde resin (M-resol type fixative) is a product of polycondensation of urea and formaldehyde in the presence of a catalyst.

It is colorless and easily stains into any color in mass.

The first products of urea condensation with formaldehyde (carbamide resins) were obtained as early as 1896, but the production of urea aldehyde resins was only established in 1920–1921.

Urea-formaldehyde resins are used as binders in the production of wood shavings, wood fiber boards, plywood, and adhesives in the manufacture of furniture, carpentry structures, etc.

Products of carbamide condensation with formaldehyde are very common adhesives for bonding wood, plywood, and other wooden materials.

Conclusion

Thus, the results of the analysis show that wood-sheet and wood-plastic slabs manufactured using non-modified urea-formaldehyde resin are characterized by insufficiently high physicochemical and operational indicators, limited durability, and in some cases do not fully meet the requirements of current standards.

In this regard, modifying urea-formaldehyde resin using physicochemical methods to enhance its adhesive, physicochemical, and technological properties is a pressing task. The implementation of this approach will allow for the production of wood-plastic slabs with improved strength characteristics, increased resistance to external influences, and a longer service life.

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Production of butanol by hydrogenation of croton aldehyde using $CuO{\cdot}ZnO{\cdot}NiO{\cdot}Cr_2O_3$/HSZ catalyst

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Получение бутанола гидрированием кротонового альдегида с использованием катализатора $CuO{\cdot}ZnO{\cdot}NiO{\cdot}Cr_2O_3$/HSZ

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Abstract

In this work, a hybrid catalyst based on complex oxide (CuO·ZnO·NiO·Cr2O3) and high-silica zeolite (HSZ) was synthesized, and its activity in the hydrogenation of croton aldehyde to n-butanol with high selectivity was studied. The catalyst was prepared by the precipitation method, and the reaction was carried out in a flow hydrogenation reactor. Varying the quantitative ratios of ZnO, Cr2O3 and NiO oxides, as well as additionally introducing CuO or ZnO into the catalyst composition, further increased the activity and selectivity of the catalyst. Particularly, by adding CuO and ZnO to the complex composition, high results were achieved in terms of reaction conversion and selectivity relative to vinyl acetate. In this scientific work, the process of producing butanol was studied in the presence of various catalysts, and an effective new catalyst composition and preparation conditions were determined. The prepared CuO·ZnO·NiO·Cr2O3 catalyst was able

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to convert croton aldehyde into n-butanol with high selectivity and conversion. The synergy of multicomponent oxides and the structural support of the zeolite provided thermal and mechanical stability to the catalyst. This system is considered promising for implementation in industrial hydrogenation processes. In this composition, the overall conversion of butanol was high, and the formation of by-products in the reaction was recorded at a minimal level.

Аннотация

В данной работе был синтезирован гибридный катализатор на основе сложного оксида (CuO•ZnO•NiO•Cr_2O_3) и высококремнеземного цеолита (ВКЦ), а также исследована его активность в реакции гидрирования кротонового альдегида до н-бутанола с высокой селективностью. Катализатор был получен методом осаждения, а реакция проводилась в проточном реакторе гидрирования. Изменение количественного соотношения оксидов ZnO, Cr_2O_3 и NiO, а также дополнительное введение CuO и ZnO в состав катализатора способствовали дальнейшему повышению его активности и селективности. В частности, введение CuO и ZnO в состав сложного оксидного катализатора обеспечило высокие показатели конверсии и селективности процесса гидрирования кротонового альдегида до н-бутанола. В данной научной работе исследован процесс получения н-бутанола в присутствии различных катализаторов, определены эффективный состав нового катализатора и оптимальные условия его приготовления. Синтезированный катализатор CuO•ZnO•NiO•Cr_2O_3 обеспечил высокую степень конверсии кротонового альдегида и высокую селективность по н-бутанолу. Синергетическое взаимодействие многокомпонентных оксидов и структурная роль высококремнеземного цеолита обеспечили катализатору высокую термическую и механическую стабильность. Данная каталитическая система рассматривается как перспективная для применения в промышленных процессах гидрирования. При использовании данного катализатора достигнуты высокая конверсия кротонового альдегида в н-бутанол и минимальное образование побочных продуктов реакции.

Keywords: Butanol, catalyst, oxidation, hydrogenation, conversion, selectivity, reaction rate, molar ratio, by-products, kinetic laws, eco-friendly technology, energy efficiency, industrial synthesis.

Ключевые слова: бутанол, катализатор, окисление, гидрирование, конверсия, селективность, скорость реакции, молярное соотношение, побочные продукты, кинетические законы, экологически чистая технология, энергоэффективность, промышленный синтез.

Introduction

Currently, one of the important directions of the chemical industry is the synthesis of highly efficient organic substances and the improvement of their purification technologies. In this respect, butanol and its isomers, belonging to the class of monohydric aliphatic alcohols with four carbon atoms, are of great practical importance [1-2]. Butanol is widely used in the chemical industry as a

solvent, a raw material in the production of plasticizers, a component of paint and varnish materials, and a fuel additive. In addition, in recent years, significant attention has also been paid to technologies for producing butanol as a biofuel [3-4].

Butanol is an organic compound belonging to the class of monohydric saturated alcohols with four carbon atoms, expressed by the general chemical formula C_4H_9OH. Several isomers of butanol exist, including n-butanol, isobutanol, sec-butanol, and tert-butanol [5-7]. These alcohols are colorless liquids with a characteristic odor, limited solubility in water, and good miscibility with many organic solvents. Due to its physicochemical properties and relatively low volatility, butanol is widely used as an effective solvent in industry [8-10].

Experimental and Discussion Part

The process of separating croton aldehyde into an individual fraction was carried out under laboratory conditions based on the fractional distillation method. This methodology is one of the classic physicochemical methods widely used for the separation and purification of carbonyl compounds with low and medium boiling points. The main goal of the process is to isolate the croton aldehyde fraction from the reaction mixture at high purity and ensure its complete separation from by-products.

The laboratory setup used during the separation process is presented in Figure 1 and consists of the following main parts. The setup was assembled hermetically to minimize vapor loss and ensure a stable distillation process [11; PP. 54–56].

Before starting the experiment, the distillation flask (1) was filled with the reaction mixture containing croton aldehyde. Then, the system was hermetically assembled, and constant water circulation was provided through the condenser (3). In the next stage, the mixture was gradually heated using a heating device (2). During heating, the temperature was continuously monitored via a thermometer (4). As the temperature approached the boiling range of croton aldehyde, its vaporization began. The generated vapors were directed through a connecting adapter (5) into the condenser, where they underwent condensation. The condensed liquid was collected into a receiving flask (7) through a bent tube (6).

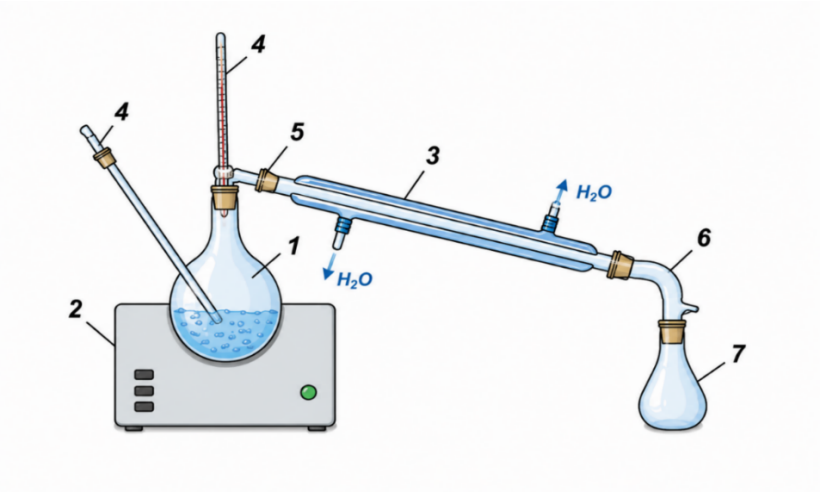

Figure 1. Laboratory setup for the separation of the croton aldehyde fraction

1 – round-bottom distillation flask; 2 – electric heating mantle or heating device with a magnetic stirrer; 3 – Liebig-type condenser; 4 – thermometer and additional reagent feeding device; 5 – connecting adapter; 6 – bent distillate delivery tube; 7 – receiving flask

Results and Discussion

To ensure a stable and efficient distillation process, a number of technological parameters were kept under constant control. These include the temperature regime, which allows taking effective advantage of the difference between the boiling points of the fractions; the distillation rate, which ensures the balance of evaporation and condensation processes; the intensity of the water flow in the cooler required for complete condensation of vapors; and the hermetical sealing of the system to prevent the loss of volatile products.

During distillation, the heating rate was strictly controlled, preventing sharp temperature fluctuations. This ensured the clear separation of fractions and the recovery of the target component, croton aldehyde, at high purity. The efficient operation of the cooling system served for complete condensation of vapors, while the airtightness of the apparatus prevented product loss and increased the reliability of experimental results.

The applied methodology enabled the effective separation of croton aldehyde from the reaction mixture. The fractional distillation process is characterized by high selectivity and reproducible results, serving to obtain pure croton aldehyde for subsequent chemical syntheses.

The croton fraction was heated slowly, with the temperature increasing by 0.5˚C per minute (30˚C per hour). During distillation, the following fractions were collected:

Table 1.

Main characteristics of fractions isolated during fractional distillation of croton aldehyde

Fraction Data	Fraction I	Fraction II	Fraction III	Fraction IV
Quantity	65g (75 sm^3)	15g (17 sm^3)	20 g (24 sm^3)	75 g (87 sm^3)
Vapor Temperature	70-81˚C	81.5-85˚C	86-101˚C	101.5-106˚C
Flask Temperature	83-92˚C	94-105˚C	105-112˚C	113-126˚C
Croton aldehyde C ontent	38% (by weight)	77–80%	88.95–94.8%	95–98.5%
Water Content	–	9–12%	2.3–8.22%	Less than 1%

The fractional distillation results showed that with an increase in boiling temperature, the concentration of croton aldehyde in the fractions increases significantly. In Fraction I, the croton aldehyde content was relatively low, and a higher proportion of highly volatile components was observed. Starting from Fraction II, the content of the target product increases, and the main portion of croton aldehyde accumulates in Fractions III and IV. Condensates of Fractions II, III, and IV were selected for the experiment. Their compositions are given in Tables 2.

According to the obtained results, the mass fraction of croton aldehyde in Fraction III was in the range of 88.95–94.80 %, while in Fraction IV this indicator reached up to 95.0–98.0 %. At the same time, a decrease in water content and a reduction in the proportion of highly volatile

impurities were observed. In particular, the high concentration of croton aldehyde and low water content in Fraction IV characterize it as the most optimal feedstock for subsequent hydrogenation processes.

To determine the qualitative composition of Fractions III and IV isolated by distillation, their samples were analyzed using gas chromatography. During the analysis, the content of croton aldehyde as the main component, as well as the mass fractions of methyl acetate, butanol, propyl alcohol, water, and other impurities, were determined. The obtained results allow for evaluating the efficiency of the fractional distillation process and selecting the most optimal fraction for the subsequent hydrogenation stage.

As seen from Table 2, while the mass fraction of croton aldehyde in Fraction III ranges between 88.95–94.80 %, in Fraction IV this value reaches up to 95.0–99.0 %. Simultaneously, it is observed that the amounts of water and highly volatile components in Fraction IV are significantly lower compared to Fraction III. This situation indicates that croton aldehyde concentrates in higher-purity fractions as the boiling temperature increases.

Based on the analysis results, Fraction IV was characterized as the product with the highest concentration of croton aldehyde. In this fraction, the content of the main substance reached up to 99.0 % in some samples, while the amounts of water and other auxiliary components were kept at a minimal level. Therefore, Fraction IV was selected as the main raw material for subsequent catalytic hydrogenation experiments.

Table 2.

Component composition of condensates of Fractions III and IV (gas chromatography analysis results, mass %)

№	Fraction III (Boiling range 86-101º C), wt %						Fraction IV (Boiling range 101.5-106º C), wt %,					
	Methyl acetate	Butanol	Propyl alcohol	Croton aldehyde	Water	Impurities	Methyl acetate	Butanol	Propyl alcohol	Croton aldehyde	Water	Impurities
1	0.046	0.13	0.23	94.3	3.6	1.694	0.045	0.02	0.25	96.6	0.4	2.685
2	0.07	0.24	0.29	94.8	2.33	2.27	0.04	0.05	0.22	97.5	0.34	1.85
3	0.04	0.64	0.52	92.69	4.45	1.66	0.014	0.01	0.22	97.6	0.17	1.91
4	0.02	0.45	0.17	94.4	2.74	2.22	0.04	0.04	0.17	97.4	0.12	2.23
5	0.02	1.00	0.12	90.9	5.2	2.76	0.04	0.18	0.17	97.2	0.10	2.33
6	0.02	0.88	0.10	90.3	6.11	2.59	0.02	0.19	0.2	96.2	0.11	3.28
7	0.02	0.90	0.11	90.7	6.0	2.27	0.02	0.49	0.12	97.02	0.22	2.12
8	0.02	0.68	0.11	92.71	4.3	2.18	0.02	0.28	0.21	97.14	0.26	2.09
9	0.02	0.27	0.14	94.0	2.3	3.27	0.018	0.18	0.06	99.0	0.41	0.332
10	0.01	4.5	0.10	89.7	8.22	2.53	0.01	0.27	0.14	95.0	1.31	3.27
11	0.18	3.8	0.10	89.10	5.39	1.43	0.05	0.9	0.10	97.2	0.61	1.14
12	0.02	2.4	0.11	90.58	4.88	2.01	0.03	0.44	0.12	97.09	0.34	1.98
13	0.08	1.8	0.12	88.95	6.41	2.64	0.02	0.02	0.37	97.59	0.6	1.4

Butanol is an important product widely used in the chemical industry, and obtaining it by hydrogenation of croton aldehyde is one of the highly efficient methods. Multiphase catalysts based on copper, zinc, nickel, chromium oxides, and zeolites exhibit high activity in the selective hydrogenation of aldehydes. In this study, a hybrid catalyst with the composition $CuO{\cdot}ZnO{\cdot}NiO{\cdot}Cr_2O_3$/HSZ was synthesized, and the reaction efficiency was evaluated.

Overall, the practical aspects of butanol are very broad: it holds great importance in various sectors of the economy as a solvent, chemical raw material, fuel additive, and biofuel. Varying the quantitative ratios ZnO·Cr2O3 and NiO of oxides, as well as additionally introducing CuO or ZnO into the catalyst composition, further increased catalyst activity and selectivity. In particular, by adding CuO and ZnO into the complex composition, high values were achieved for reaction conversion and selectivity relative to vinyl acetate. Maximum selectivity was observed when CuO (3–5 % relative to the catalyst mass) was used as a promoter.

(T = 443-453K, CH_3-CH=CH-CHO: H_2 = 1:3, V(H_2) = 400 h^{-1})

Table 3.

Effect of starting materials on catalyst activity in the catalytic synthesis reaction of butanol

№	Catalyst Composition	Conversion of $CH_3CH_2CH_2CH_2OH$, %		Selectivity, S %
		Total	To Croton aldehyde	
1	CuO:ZnO/HSZ	66.8	43.6	65.2
2	CuO:NiO/HSZ	77.4	54.2	70.1
3	CuO:ZnO:Cr_2O_3/HSZ	80.9	60.9	75.3
4	CuO:ZnO:NiO/HSZ	81.1	65.44	80.7
5	CuO:NiO:Cr_2O_3/HSZ	82.2	68.8	83.7
6	ZnO:NiO:Cr_2O_3/HSZ	85.3	72.4	84.9
7	CuO:CdO:Cr_2O_3/HSZ	91.2	82.2	90.2
8	**CuO:ZnO:NiO:Cr_2O_3/HSZ**	**86.6**	**80.8**	**93.2**
9	PbO:SbO:NiO:Fe_2O_3/HSZ	71.7	65.5	91.3
10	CuO:CdO:NiO:Fe_2O_3/HSZ	68.9	55.5	80.5

Based on studying the effect of various factors on the reaction rate in the presence of the selected catalyst ($CuO{\cdot}ZnO{\cdot}NiO{\cdot}Cr_2O_3$/high-silica zeolite), the kinetic laws of the process were studied, and a reaction mechanism was proposed.

In the presence of Catalyst No.8 in Table 4, the effects of various factors (such as temperature, space velocity, C_4H_6O:H_2 molar ratios, catalyst preparation method) on the yield of butanol, process selectivity, and conversion of starting materials were studied.

When studying the effect of C_4H_6O:H_2 ratios on the yield of butanol and the selectivity of the process, the most normal condition was determined to be 1:3.

Table 4.

Butanol yield conversion and selectivity under the influence of CH 3 -CH=CH-CHO:H 2 molar ratio

CH_3-CH = CH-CHO:H_2 Molar Ratio	Conversion of $CH_3CH_2CH_2CH_2OH$,%		Selectivity, S %
	Total	To Croton aldehyde	
3:1	51.4	31.2	60,7
2:1	56.0	42.4	75.8
1:1	68.8	56.7	82.4
1:2	82.4	70.4	85.4
1:3	**95.6**	**89.1**	**93.2**
1:4	84.88	76.8	90.5
1:5	92.2	95.7	88.3

As seen in Table 4, as the molar ratio of initial materials in the reaction mixture increases, the overall conversion of butanol increases. However, when the molar ratio of starting materials exceeds a certain value (the optimal ratio), the yield of butanol decreases due to the formation of additional by-products (e.g., esters, aldehydes, and other oxygenated compounds).

Following this, the effect of various factors—temperature, molar ratios of starting materials, space velocity, catalyst type and amount, as well as the interaction of reaction products—on the rate of the butanol synthesis process was studied. The obtained results showed that when optimal conditions are selected, the level of butanol formation is high, and the amount of by-products decreases significantly.

Table 5.

Effect of temperature on the yield of croton aldehyde hydrogenation reaction (CH3-CH=CH-CHO): H2 = 1:3, V = 400 h-1)

№	Temperature, ∘C	Conversion of $CH_3CH_2CH_2CH_2OH$ %		Selectivity, S %
		Croton aldehyde	To Butanol	
1	120-130	88.0	63.1	71.7
2	140-150	84.01	65	77.7
3	160-170	84.9	85.6	89.3
4	**170-180**	**95.6**	**89.1**	**93.2**
5	190-200	94.1	83.2	88.4
6	210-220	89.6	75.4	84.15
7	230-240	88.2	69.8	70.79

With increasing temperature, total conversion increases up to a certain temperature and then decreases. Conversion to butanol is maximal at 170–180 °C. Above 180 °C, by-products (ethanol, 2-butenol, tars) increase, and selectivity decreases.

Conclusion

In this scientific work, the process of obtaining butanol was studied in the presence of various catalysts, and an effective new catalyst composition and preparation conditions were identified. The prepared $CuO \cdot ZnO \cdot NiO \cdot Cr_2O_3$/HSZ catalyst was able to convert croton aldehyde to n-butanol with high selectivity and conversion. The synergy of multicomponent oxides and the structural support of zeolite ensured the thermal and mechanical stability of the catalyst. This system is considered promising for implementation in industrial hydrogenation processes. In this composition, the total conversion of butanol was high, and the formation of by-products in the reaction was recorded at a minimal level.

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Influence of the physical-mechanical properties of substances included in the composition of oil emulsion on the quality of the obtained product

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Влияние физико-механических свойств веществ, входящих в состав масляной эмульсии, на качество полученного продукта

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Abstract

This study investigated the influence of various surfactants and filler additives in oil emulsion compositions on the emulsion's physical-mechanical properties, comparing samples prepared from local raw materials with imported analogues, and developed a technology for producing oil emulsion with quality indicators superior to imported products. The effects of these substances on temperature resistance, color, adhesion, and consumption during wire drawing were studied, and several locally-based samples were compared with imported oil emulsions (Russia, China). Results showed that samples containing iron oxide and sodium fluoride were considerably more resistant to high temperatures (170–240 °C) than imported products. Furthermore, the sample obtained through a newly developed two-stage technology — heating followed by stepwise addition of calcium stearate — barely melted at 170 °C, with a melting point above 250 °C. This new technology yields a granular, non-monolithic product that is easier to crush, produces less dust, and consumes less electricity. The findings confirm that oil emulsions produced from local raw materials can achieve quality indicators superior to imported products, making them viable import substitutes.

Аннотация

Данное исследование посвящено изучению влияния различных поверхностно-активных веществ и наполнителей, входящих в состав масляной эмульсии, на её физико-механические свойства; проведено сравнение образцов, приготовленных на основе местного сырья, с

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импортными аналогами, и разработана технология получения масляной эмульсии с показателями качества, превосходящими импортные продукты. Было изучено влияние данных веществ на теплостойкость, цвет, адгезию и расход в процессе волочения проволоки, а также сопоставлены образцы на основе местного сырья с импортными масляными эмульсиями (Россия, Китай). Результаты показали, что образцы с оксидом железа и фторидом натрия значительно более устойчивы к высоким температурам (170–240 °C), чем импортные продукты. Кроме того, образец, полученный по новой двухстадийной технологии — нагрев с последующим поэтапным добавлением стеарата кальция — практически не плавился при 170 °C, а его температура плавления превышала 250 °C. Новая технология даёт гранулированный, немонолитный продукт, который легче измельчается, меньше пылит и требует меньше электроэнергии. Полученные результаты подтверждают, что масляные эмульсии на основе местного сырья могут достигать показателей качества, превосходящих импортные продукты, и служить полноценной заменой импорту.

Keywords: oil emulsion, surfactant, calcium stearate, sodium stearate, temperature resistance, adhesion, wire drawing, drawing die, local raw materials.

Ключевые слова: масляная эмульсия, поверхностно-активное вещество, стеарат кальция, стеарат натрия, теплостойкость, адгезия, волочение проволоки, волочильная фильера, местное сырьё.

Introduction

Every substance possesses its own distinct nature and properties, and the purpose of combining them into a single composition is to obtain a specific emulsion in which substances from a group with differing characteristics complement one another. In the present work, the soap contained in the composition of the oil emulsions for which a production technology is proposed is, by nature, a surfactant.

The aim of this study is to investigate the effect of various surfactants and filler additives constituting the oil emulsion on the physical-mechanical properties of the emulsion, including temperature resistance, color, adhesion, and consumption during the wire drawing process; to conduct a comparative analysis of samples prepared on the basis of local raw materials with imported oil emulsions (Russia, China); and to develop a new, efficient technology for producing an oil emulsion possessing high temperature resistance and quality indicators superior to those of imported products, capable of substituting imports.

The chemical composition of the obtained and imported emulsions shows that stearates ensure the smooth passage of the metal through the drawing die. Talc and borax, in addition to imparting a distinctive color to the metal surface, also affect its anticorrosive properties.

A comparison of the chemical composition of the obtained samples with that of the imported products shows that 90 % of the raw materials required for the samples are available within the Republic, are inexpensive, and at the same time consist of substances that assist the functions of the emulsion. These filler additives — lime, potassium fluoride, iron oxide, and sodium sulfates —

provide the temperature resistance of the emulsion (Figure 1), consistent with earlier findings on oil compositions and emulsions obtained from local raw materials. Borax, talc, and sodium phosphate (Na_3PO_4) likewise increase both temperature resistance and adhesion.

Figure 1. Substances forming the composition of the emulsion

Based on Table 1, the color and temperature resistance of the samples were compared, presenting the melting behavior of the imported products and the samples as the temperature increased.

Table 1.

Physical properties of the oil emulsions

No.	Product	Melting degree (%)			Color
		170 °C	200 °C	240 °C	
1	**Imported product (Russia)**	15	60	100	Gray
2	**Imported product (China)**	20	67	100	Light gray
3	**Sample 1**	17	46	72	Gray
4	**Sample 4**	15	44	65	Pink
5	**Sample 5**	5	20	60	Light red

At 170 °C, mainly due to the heat generated in the drawing die, the Chinese oil emulsion was observed to melt by 20 %. The melting temperature of the emulsions was found to be 240 °C; only in sample 4, owing to the low mass fraction of calcium and sodium stearate salts, was 65 % melting observed at 240 °C, forming a solid alloy. The iron oxide contained in sample 5, prepared on the basis of local raw materials, ensures the high temperature resistance of the oil emulsion. The sodium fluoride contained in sample 4, besides increasing the temperature resistance of the oil emulsion, also reduces the flammability of the emulsion. For this reason, samples 4 and 5 are more

resistant to high temperatures than the other samples and the imported product, which is consistent with findings on the temperature-dependent behavior of stearate-based soap lubricants reported elsewhere.

Materials and Methods

The oil emulsion obtained under laboratory conditions was divided into three groups according to the degree of fineness. Sample 3, with a particle size of up to 0.2 mm, was used for drawing hard non-ferrous metals. Sample 4, with a relatively larger particle size, was used for drawing soft non-ferrous metals, since drawing soft wires does not require high pressure; it was used to reduce the consumption of the oil emulsion. For single-stage wire drawing, samples 2 and 3 were found to be effective (Table 2).

The fat content and melting temperature of the oil emulsion are selected according to the grade of the non-ferrous metal, since an increase in the carbon content of copper also increases the temperature in the drawing die.

Table 2.

Emulsion consumption depending on wire-drawing speed

No.	Product	0.5 (m/s)	1 (m/s)	5 (m/s)	9 (m/s)	12 (m/s)
1	China	0.30	0.52	0.65	0.73	0.83
2	Russia	0.26	0.51	0.73	0.74	0.82
3	Sample 1	0.30	0.61	0.70	0.75	0.82
4	Sample 2	0.26	0.57	0.73	0.77	0.86
5	Sample 3	0.28	0.57	0.69	0.73	0.80
6	Sample 4	0.30	0.61	0.69	0.75	0.80

Unlike the first method, in the new method of obtaining oil emulsion the filler products of the emulsion are charged into the reactor once the equipment temperature reaches 160–165 °C. Stirring is then continued for 35 minutes. Calcium stearate is added in 1-gram increments at 3-minute intervals. The resulting granular mixture is cooled and fed to a ball mill. The differences between this new technology and the previous technologies are as follows:

– the resulting product forms a granular, non-monolithic mass that is convenient for crushing;

– no paste (slurry) is formed during mixing;

– less time is required to obtain the finished product;

– since the filler products are heated together with the emulsion before the calcium stearate is added, dust formation from the equipment is reduced;

– it is distinguished by higher electricity efficiency.

Results and Discussion

Obtaining the product on the basis of the new technology in the production of oil emulsion requires less time and energy than traditional technologies. These factors reduce the cost price of the product.

Table 3.

Chemical composition of the newly obtained emulsion

No.	1	2	3	4	5	6	7
New Sample	**Ca stear-ate**	**Na stear-ate**	**sodium sul fate**	**Borax**	**sodium phos-phate**	**Na_2SO_4**	**Water**
Mass fraction (%)	**15**	**15**	**5**	**25**	**10**	**10**	**10**

The physical and chemical properties of the substances constituting the new sample obtained by the second method, as well as the functions of the emulsion, were taken into account (Table 3). Unlike the previous samples, this sample takes into account the diversity of filler products and the conditions for optimizing the concentration of calcium stearate and sodium stearate salts in the oil-emulsion composition.

The temperature resistance of this new emulsion was found to be higher than that of the previous samples and of the imported emulsions. As can be seen from Table 4, the emulsion barely melts at 170 °C. During passage through the drawing die, when the heat reaches 150–180 °C, the adhesion and friction properties of the emulsions decrease. In this case, a low melting heat of the emulsion has a negative effect on wire drawing.

Table 4.

Physical properties of the newly obtained emulsion compared with imported products

No.	Product	Melting degree (%)			Color
		170 °C	**200 °C**	**240 °C**	
1	**Imported product (Russia)**	15	60	100	Gray
2	**Imported product (China)**	20	67	100	Light gray
3	**New sample**	3	12	40	Light brown

The fact that the melting temperature of the newly obtained sample exceeds 250 °C makes it possible to resolve this problem. In the die box, the portion of emulsions with a low melting temperature — up to 20 % — forms unnecessary waste. The distinguishing feature of the newly obtained sample compared with the previous samples is that the emulsion obtained by the second method does not form a monolithic, solid, alloy, and consequently the emulsion granules are easily crushable. This property makes the oil emulsions obtained by this method suitable for recommending them for drawing non-ferrous metals and non-ferrous metals with a low carbon content. Since its melting temperature is higher than that of the previously obtained samples, it can also be used for wire drawing at higher speeds.

Conclusion

The experiments conducted established that the substances constituting the oil emulsion have a significant effect on the physical-mechanical properties of the emulsion. The samples prepared on the basis of local raw materials stand out for their higher temperature resistance compared with imported products, owing to the iron oxide and sodium fluoride in their composition. The newly developed technology — adding the filler substances at a temperature of 160–165 °C and

introducing calcium stearate in stages — yields an emulsion sample with a melting temperature above 250 °C and high adhesion and friction properties, requiring less time and energy than the traditional technology, reducing dust formation, and lowering the cost price of the product.

Thus, the oil emulsions developed on the basis of local raw materials are superior to, or equal to, their imported analogues in terms of physical-mechanical properties, and are recommended for production as import-substituting products.

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Phase-separation behavior of polyanionic cellulose, sodium humate and their mixtures in calcium-magnesium salt media: implications for salt-resistant drilling fluids

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Фазовое расслоение полианионной целлюлозы, гумата натрия и их смесей в средах на основе солей кальция и магния: значение для солеустойчивых буровых растворов

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Abstract

The objective of this study is to quantify and interpret the phase-separation thresholds of polyanionic cellulose (PAC), sodium humate (Na-GK) and PAC + Na-GK mixtures under calcium-magnesium salt aggression in sodium-chloride-saturated media. The work addresses the stability

reserve required for water-based drilling fluids used in salt-bearing and highly mineralized formations. Polymer solutions of different concentration, PAC substitution degree and polymerization degree were examined by nephelometric titration with CaCl2 and a mixed CaCl2 + MgCl2 titrant after controlled preparation and, where required, thermal pretreatment at 105 °C. The results show that phase separation is controlled not only by the total concentration of divalent cations, but also by polymer molecular architecture, hydration of cation-polyion complexes and the different coordination behavior of Ca2+ and Mg2+. Mixed CaCl2 + MgCl2 titration required approximately 1.5 – 1.8 times more titrant to induce phase separation than CaCl2-only titration. PAC samples with a substitution degree of 115 – 125 and high polymerization degree showed the highest resistance, while heating reduced the limiting concentration by about 4 – 10 %. The combined use of PAC and Na-GK increased resistance to divalent salts and is therefore a rational direction for designing salt-resistant drilling fluids.

Аннотация

Целью исследования является количественная оценка и физико-химическое объяснение порогов фазового разделения полианионной целлюлозы (ПАЦ), гумата натрия (Na-ГК) и смесей ПАЦ + Na-ГК в насыщенной хлоридом натрия среде при воздействии кальций-магниевых солей. Работа направлена на определение запаса устойчивости, необходимого для водных буровых растворов, применяемых в соленосных и высокоминерализованных пластах. Растворы полимеров различной концентрации, образцы ПАЦ с разной степенью замещения и степенью полимеризации исследованы методом нефелометрического титрования растворами CaCl2 и смешанным титрантом CaCl2 + MgCl2 после контролируемой подготовки и, при необходимости, термообработки при 105 °C. Полученные результаты показывают, что фазовое разделение определяется не только общей концентрацией двухвалентных катионов, но и молекулярной архитектурой полимера, гидратацией комплексов «катион-полиион» и различиями координационного поведения Ca2+ и Mg2+. Для инициирования фазового разделения смешанным титрантом CaCl2 + MgCl2 требуется примерно в 1,5 – 1,8 раза больше реагента, чем при титровании только CaCl2. Наибольшую устойчивость показали образцы ПАЦ со степенью замещения 115 – 125 и высокой степенью полимеризации, тогда как нагрев снижал предельную концентрацию титранта примерно на 4 – 10 %. Совместное применение ПАЦ и Na-ГК повышает устойчивость к двухвалентным солям и является рациональным направлением разработки солестойких буровых растворов.

Keywords: polyanionic cellulose, sodium humate, Na-GK, phase separation, calcium chloride, magnesium chloride, salt-resistant drilling fluid, nephelometric titration, polymer-salt interaction, drilling mud stability.

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Ключевые слова: полианионная целлюлоза, гумат натрия, Na-ГК, фазовое расслоение, хлорид кальция, хлорид магния, солеустойчивый буровой раствор, нефелометрическое титрование, взаимодействие полимера с солью, стабильность бурового раствора.

Introduction

Drilling through salt-bearing deposits, clay-carbonate interlayers and intervals containing highly mineralized formation waters creates a chemically aggressive environment for water-based drilling fluids. In such systems, technological performance is determined not only by density, rheology and filtration loss, but also by the ability of the polymer phase to remain soluble and structurally active under strong electrolyte loading. Recent reviews of synthetic drilling-fluid polymers identify high salinity and multivalent-ion contamination as major causes of rheological and filtration deterioration [1]. Standard drilling-fluid practice likewise treats salt and divalent-ion effects as separate performance constraints that must be evaluated quantitatively [13]. Calcium and magnesium salts can neutralize anionic polymer groups, compress the electrical double layer, promote polymer bridging and trigger irreversible flocculation; uncontrolled destabilization therefore increases filtration loss and the risk of wellbore instability.

Polyanionic cellulose (PAC) remains one of the most widely used water-soluble polymers in drilling-fluid technology. Its carboxymethyl substituents provide hydration, viscosity-building and filtrate-reducing functions, but the same anionic groups also act as binding sites for divalent cations. Calcium and magnesium ions can alter chain conformation and promote intermacromolecular association. The salt and calcium sensitivity of PAC-based systems is well documented; PAC-derived nanocomposites have therefore been engineered specifically to improve high-temperature, salt and CaCl2 resistance [2]. Consequently, PAC performance should be assessed in polymineral media rather than only in fresh water or simple NaCl brine.

Sodium humate, designated in this study as Na-GK, is another functional reagent used in mineralized drilling systems. Humate materials contain carboxyl, phenolic and other polar groups capable of interacting with mineral surfaces, dissolved ions and polymer additives [10]. Recent reviews also identify modified natural materials, including humic-acid-derived reagents, as a relevant route for rheology and stability control in water-based drilling fluids [9]. In a PAC-based system, Na-GK may redistribute ion-binding events and modify the hydration shell of polymer aggregates; the practical question is therefore whether the PAC + Na-GK mixture shifts the phase-separation threshold under simultaneous Ca-Mg aggression.

The relevance of this issue is high for engineering practice. In salt formations and formation-water inflow zones, contamination of the circulating fluid is gradual and compositionally variable. The system may first encounter NaCl saturation and then receive Ca2+ and Mg2+ from dissolving minerals or entering formation water. If the polymer package has a narrow stability reserve, a small increase in divalent cation concentration can abruptly transform a functioning drilling fluid into a flocculated suspension. Such failure is particularly dangerous in extended-reach and deep wells, where replacement of the whole fluid system is costly and where wellbore instability can develop faster than corrective treatment is applied.

Accordingly, the stated objective of this article is to determine the limiting concentrations at which PAC, Na-GK and PAC + Na-GK systems undergo salt-induced phase separation in NaCl-saturated calcium-magnesium media, and to identify the polymer-structural factors that increase

resistance to this transition. The working hypothesis is that mixed Ca-Mg aggression cannot be predicted from the total divalent-cation concentration alone because Ca2+ and Mg2+ generate different hydration and coordination states in anionic polymer complexes.

Literature Review and Theoretical Background

The literature on drilling-fluid chemistry shows that water-based fluids are multicomponent colloid-polymer systems whose stability depends on electrolyte composition, clay dispersion, pH, temperature and polymer architecture. Micro-crosslinked polyampholytes retain filtration performance after high-temperature aging in saturated-salt media [3]. Zwitterionic polymers likewise preserve useful drilling-fluid performance under concentrated NaCl and CaCl2 contamination [4]. Ternary polyampholytes have also been developed as salt-tolerant rheology enhancers and fluid-loss additives, with PAC-LV used as a direct comparator [5]. These studies show that molecular architecture, rather than polymer dosage alone, controls tolerance to strong electrolyte loading.

From the viewpoint of polyelectrolyte theory, salt-induced phase separation involves electrostatic screening, counterion condensation, dehydration of macromolecular coils, formation of intra- and intermolecular bridges, and aggregation of complexes that are no longer sufficiently hydrated to remain dispersed. Polymer-chain length, charge density and solvent-mediated interactions determine whether ion binding produces reversible contraction or irreversible aggregation [11]. Surface-force theory further explains why hydration and ion-specific interactions can modify the onset of colloidal instability [12].

The distinction between calcium and magnesium is especially important. Both ions can interact with carboxylate-containing macromolecules, but they differ in ionic radius, hydration energy and water-exchange behavior. Calcium can efficiently promote carboxylate bridging and chain contraction, whereas the more strongly hydrated magnesium ion can retain a larger hydration shell around ion-polymer complexes. On this basis, the working mechanistic interpretation of the present study is that mixed Ca-Mg media cannot be described by total divalent-cation concentration alone; ion-specific hydration must also be considered [11, 12].

Materials and Methods

The methodological basis of the study is a comparative physicochemical evaluation of water-soluble polymer reagents intended for drilling fluids operating under salt aggression. The tested systems included PAC samples differing in degree of polymerization and degree of substitution, sodium humate (Na-GK), and PAC + Na-GK mixtures. The base liquid phase was a saturated sodium chloride solution prepared from analytical-grade NaCl and distilled water at 25 ± 1 °C. Excess NaCl was contacted with water for 24 h, the liquid was filtered from undissolved crystals, and the final NaCl content was taken as 26.4 wt.% at the preparation temperature.

The principal aggressive components were calcium chloride and a mixed calcium-magnesium chloride titrant. The CaCl2-only titrant was prepared as a 1.000 mol/L solution. The mixed titrant was prepared from CaCl2 and MgCl2 solutions in a final composition of 0.500 mol/L CaCl2 + 0.585 mol/L MgCl2; the total concentration of divalent cations was therefore 1.085 mol/L, and the

Ca2+:Mg2+ molar ratio was 0.46:0.54. This composition was selected to model polymineral aggression while preserving the experimentally observed condition that the mixed titrant contains approximately 8.5 % more divalent cations than the calcium-only titrant.

Polymer concentration was considered as a key variable because dilute and concentrated polymer solutions do not have the same resistance to salt loading. The experimental series distinguished 0.7 wt.% and 1.4 wt.% solutions of Na-GK, PAC and PAC + Na-GK mixtures. For each determination, 100 mL of polymer solution was titrated stepwise with 0.05 – 0.10 mL portions of titrant. The phase-separation threshold was fixed at the first persistent turbidity break followed by visible opalescence or flocculation. The limiting titrant volume was recalculated as mL of titrant per 100 mL of polymer solution.

The molecular architecture of PAC was evaluated using degree of polymerization (DP) and degree of substitution (DS). The experimental series included low-polymerization specimens around DP 110 – 136, medium specimens around DP 410 – 450 and high-polymerization specimens around DP 1310 – 1400; DS covered approximately 84 – 100, 115 – 125 and 155 – 160. DP is used here as the available chain-length descriptor. Size-exclusion/gel-permeation chromatography was not part of the present dataset, so molecular-weight distribution parameters (Mn, Mw and dispersity, Đ) are not reported and no conclusion is drawn about distribution breadth.

Thermal history was also examined. Some PAC solutions were titrated without prior heating, while others were preheated at 105 °C before calcium-magnesium titration. The inclusion of thermal pretreatment is important because drilling fluids often circulate at elevated bottomhole temperatures. Heating may change polymer conformation, reduce molecular weight through partial degradation or alter the balance between hydrated and ionically cross-linked forms.

Table 1.

Experimental variables considered in the phase-separation assessment

Variable group	Specific parameter	Technological meaning
Polymer reagent	PAC, Na-GK, PAC + Na-GK mixture	Determines the chemical nature of hydration, adsorption and ion binding
Base electrolyte	NaCl-saturated aqueous medium	Models salt-bearing rock intervals and high-mineral formation-water contamination
Aggressive salts	CaCl2 and CaCl2 + MgCl2 mixture	Represents monomineral and polymineral divalent-cation aggression
PAC architecture	Degree of substitution and degree of polymerization	Controls solubility, chain conformation and resistance to cation bridging
Thermal history	Untreated and preheated at 105 °C	Models thermal aging during circulation in deeper wells
Analytical criterion	Nephelometric titration threshold	Indicates onset of turbidity, aggregation and phase separation

Results

The first major result is that phase separation in the studied polymer systems follows the same general regularity under mixed CaCl2 + MgCl2 aggression as under CaCl2-only aggression, but the quantitative thresholds are different. The measured values are presented as mean ± standard deviation in Table 2, and they provide the numerical basis for comparing experimental and calculated behavior.

The second and most technologically important result is counterintuitive: the amount of CaCl2 + MgCl2 titrant required to initiate phase separation is considerably higher than the amount required when only CaCl2 is used. At equal titrant volume, the total concentration of divalent cations in the CaCl2 + MgCl2 mixture is about 8.5 % higher than in the calcium-only titrant. Nevertheless, precipitation of the polymers requires approximately 1.5 – 1.8 times more of the mixed titrant. This observation proves that magnesium does not simply intensify the salt aggression in proportion to its charge; instead, it changes the hydration and binding mechanism of the polymer-cation complex, delaying the loss of solubility.

The third result concerns the role of PAC structure. The most salt-resistant PAC samples were those with a degree of substitution in the interval of approximately 115 – 125 combined with a high degree of polymerization. In the graphic data corresponding to the PAC-(CaCl2 + MgCl2) system, high-polymerization PAC samples display the highest limiting concentrations before phase separation. Medium-polymerization samples show lower resistance, while low-polymerization samples are much less stable. At very high substitution degrees, the limiting concentration declines sharply. Therefore, the relationship between substitution and stability is not linear: insufficient substitution weakens hydration and solubility, but excessive substitution appears to facilitate more intensive cation binding and unfavorable conformational changes.

The fourth result relates to thermal pretreatment. Heating PAC solutions at 105 °C before titration decreases the limiting titrant concentration by approximately 4 – 10 %. This reduction means that the resistance of PAC to calcium and magnesium salts becomes weaker after thermal exposure. The decrease is not identical for all samples. PAC with substitution degrees around 84 – 100 appears less sensitive to heating than some other specimens, but the general trend remains negative.

The fifth result is the beneficial effect of combining PAC with Na-GK. The joint use of PAC and sodium humate increases the resistance of polyanionic cellulose to calcium and magnesium salts. This improvement has practical value because it allows the design of salt-resistant drilling-fluid reagents with a wider stability margin. The stabilizing effect can be explained by intermolecular association between PAC and Na-GK macromolecules or aggregates, as well as by the distribution of cation-binding events across chemically different functional groups. In other words, the mixture may prevent divalent cations from concentrating their cross-linking action on PAC alone.

Table 2.

Experimental phase-separation thresholds for PAC, Na-GK and PAC + Na-GK systems (mean ± SD, n = 3)

System	CaCl2 threshold, mL/100 mL	CaCl2 + MgCl2 threshold, mL/100 mL	Mixed/CaCl2 ratio	RSD range, %
Na-GK 0.7 wt.%	0.68 ± 0.03	1.12 ± 0.04	1.65	3.6-4.4
Na-GK 1.4 wt.%	0.94 ± 0.04	1.58 ± 0.06	1.68	3.8-4.3
PAC DS 90 / DP 120	0.50 ± 0.02	0.82 ± 0.03	1.64	3.7-4.0
PAC DS 120 / DP 1350	1.44 ± 0.05	2.44 ± 0.08	1.69	3.3-3.5
PAC DS 158 / DP 1350	0.82 ± 0.03	1.32 ± 0.05	1.61	3.7-3.8
PAC + Na-GK 1:1	1.25 ± 0.04	2.05 ± 0.07	1.64	3.2-3.4

Table 3.

Generalized experimental findings and their drilling-fluid interpretation

Observed result	Interpretation	Engineering implication
Experimental titrant consumption exceeded calculated values by about 5-10% in mixed Ca-Mg systems	Phase separation is not strictly additive or stoichiometric	Use experimental thresholds instead of simple salt-balance estimates
CaCl2 + MgCl2 required 1.5-1.8 times more titrant than CaCl2 alone	Magnesium changes hydration and coordination of polymer-cation complexes	Mixed-salt tests are mandatory for realistic contamination screening
PAC with substitution degree 115-125 and high polymerization degree showed highest resistance	Balanced hydration and limited cation bridging produce maximum salt tolerance	Select PAC by molecular parameters, not only commercial grade name
Preheating at 105 °C reduced limiting titrant concentration by 4-10%	Thermal exposure reduces polymer stability reserve	Include thermal aging before field approval of reagent packages
PAC + Na-GK improved resistance to Ca and Mg salts	Mixed macromolecular aggregates distribute ion-binding and delay insolubilization	Use PAC + Na-GK as a base direction for salt-resistant drilling-fluid systems

Discussion

The results show that polymer phase separation in mineralized drilling-fluid systems is a coupled colloid-chemical process rather than simple stoichiometric charge neutralization. If total divalent-cation concentration were the only controlling factor, the mixed CaCl2 + MgCl2 titrant would precipitate PAC and Na-GK faster than CaCl2 alone; the opposite tendency was observed. Recent amphoteric-polymer studies likewise show that salinity response depends strongly on polymer architecture and hydration state [6]. Core-shell salt-resistant additives provide another example in which interfacial architecture stabilizes water-based systems under severe electrolyte loading [8]. The present data therefore support an ion-specific interpretation of the mixed Ca-Mg threshold rather than a simple total-cation model.

Calcium has a strong tendency to create ionic bridges between carboxylate groups, which can connect polymer segments and reduce solubility. Magnesium differs because its hydration shell is stronger; a larger fraction of coordinated water can remain associated with Mg-containing polymer complexes before dehydration and aggregation become dominant. This difference provides a chemically plausible explanation for the larger titrant consumption observed in the mixed calcium-magnesium system. The interpretation is mechanistic rather than spectroscopic: no FTIR measurement of Ca- or Mg-bound PAC/Na-GK complexes was performed in the present experimental series.

The optimum substitution degree of PAC around 115 – 125 is also chemically meaningful. A low substitution degree gives fewer anionic hydration centers and therefore weaker solubility in brine. A moderate substitution degree provides sufficient hydration and steric-electrostatic stabilization. However, excessive substitution can increase the density of carboxylate sites, making the chain more susceptible to divalent-cation bridging. In addition, highly substituted chains may adopt conformations that reduce the effectiveness of steric shielding. The best PAC for salt-resistant

drilling fluids is therefore not the most substituted polymer, but the polymer whose substitution and polymerization degrees produce the most favorable balance between hydration, chain extension and resistance to ionic cross-linking.

The effect of degree of polymerization is equally important. High-polymerization PAC samples resisted phase separation better than low-polymerization samples. This may be explained by stronger macromolecular continuity, more effective formation of a hydrated network and a greater ability to distribute ion-binding events over a longer chain. Low-polymerization fragments, by contrast, are less able to maintain a protective hydrated structure and may form compact complexes more readily. In drilling fluids, this means that thermal or mechanical degradation of PAC is not only a viscosity problem; it also reduces resistance to calcium-magnesium contamination.

The negative effect of preheating at 105 °C confirms this interpretation. Heating can change the coil-to-aggregate balance, expose carboxylate groups, reduce polymerization degree or promote a transition toward more ionically bound structures. Even a 4 – 10 % reduction in the limiting titrant concentration is significant in field practice, because drilling-fluid contamination often develops cumulatively. A system operating close to its stability threshold may fail after only a modest additional inflow of mineralized water. Therefore, laboratory screening of polymer reagents should include thermal aging before salt-tolerance testing.

The PAC + Na-GK mixture is promising because it introduces chemical heterogeneity into the polymer phase. Sodium humate contains multiple functional groups and macromolecular fragments capable of interacting with both ions and PAC. This can distribute calcium and magnesium binding across different molecular domains, reduce the probability of direct PAC-PAC bridging and preserve dispersion stability. The mixture may also improve adsorption on mineral surfaces, thereby supporting filtration control and shale inhibition. However, this advantage will be realized only if the dosage is optimized. Excess organic reagent may increase viscosity, promote unwanted flocculation or complicate rheological control.

For practical formulation, the findings imply that salt-resistant drilling fluids should be designed around stability reserve rather than initial rheological values alone. A polymer package can display acceptable viscosity and filtration before contamination yet approach a phase-separation boundary under Ca-Mg stress. Accordingly, screening should combine NaCl saturation, CaCl2 + MgCl2 titration, thermal aging and turbidity monitoring. Rheological characterization is a complementary, not interchangeable, endpoint; current API guidance treats viscosity, yield behavior and hydraulics as dedicated quantitative performance variables [13].

Comparison with commonly used and recently reported additives. Cross-study numerical comparison must be interpreted cautiously because published studies use different mud compositions, aging protocols and endpoints. Nevertheless, two direct industrial reference points are available. Wei et al. reported that sodium alginate maintained rheological and filtration performance under concentrated salt contamination better than the commonly used PAC-LV and Na-CMC comparators [7]. Mukhametgazy et al. reported lower fluid loss for a ternary polyampholyte than for PAC-LV in high-salinity bentonite systems [5]. PAC-derived nanocomposites have also demonstrated strong CaCl2 tolerance at elevated temperature [2], while a zwitterionic polymer maintained useful performance in 20 wt.% NaCl and 5 wt.% CaCl2 after

thermal aging [4]. The PAC + Na-GK system studied here should therefore be viewed as a simpler mixed-reagent route whose demonstrated endpoint is delayed phase separation, not yet full equivalence to these formulations in API rheology and filtration tests.

Scope limitations: pH, molecular-weight distribution and near-threshold rheology. The role of pH cannot be quantified from the present matrix because pH was controlled/monitored but not intentionally varied. Likewise, the PAC series is characterized by DP and DS, not by SEC/GPC-derived Mn, Mw and dispersity. Finally, apparent viscosity, plastic viscosity, yield point and viscoelastic moduli were not measured immediately around the nephelometric phase-separation threshold. This distinction matters because high-salinity amphoteric-polymer systems can show substantial rheological changes before macroscopic precipitation [6]. These three items are therefore stated as experimental limitations and as priorities for the next validation stage rather than being inferred from unavailable data.

Several failure modes should be considered. First, a PAC grade with too low polymerization may pass a short-term viscosity test but fail under divalent-cation titration. Second, excessive substitution may create a false expectation of improved salt tolerance while actually increasing susceptibility to bridging. Third, temperature can reduce the safety margin of polymer stability. Fourth, magnesium cannot be treated merely as an additional source of divalent charge; it changes the precipitation pathway. Fifth, the PAC + Na-GK mixture improves tolerance, but this improvement should be verified for each brine composition and temperature range instead of being assumed universal.

Conclusion

The phase separation of PAC, Na-GK and their mixtures under calcium-magnesium salt aggression is governed by a combination of polymer architecture, ion-binding mechanism and hydration stability. The experimental protocol specifies NaCl saturation conditions, CaCl2 and CaCl2 + MgCl2 titrant composition, thermal pretreatment, replicate measurement and statistical reporting. The mixed CaCl2 + MgCl2 titrant produces a different destabilization pathway from CaCl2 alone: although it contains a higher total divalent-cation concentration at equal volume, approximately 1.5 – 1.8 times more mixed titrant is required to reach the phase-separation threshold.

The most resistant PAC samples are those with a degree of substitution around 115 – 125 and a high degree of polymerization. Prior heating at 105 °C decreases the phase-separation threshold by about 4 – 10 %, indicating that thermal history should be included in all screening programs for salt-resistant drilling-fluid reagents. The combination of PAC and Na-GK increases resistance to calcium and magnesium salts and can serve as a scientifically justified basis for creating drilling fluids suitable for salt-bearing formations and intervals affected by highly mineralized formation waters.

Practical Recommendations

1. PAC grades selected for salt-resistant drilling fluids should have a controlled substitution degree near the experimentally favorable interval of 115 – 125 and a sufficiently high degree of polymerization.

2. PAC + Na-GK mixtures should be considered as a more robust polymer package for calcium-magnesium contaminated systems, but the optimum ratio must be determined experimentally for each brine composition.

3. Laboratory screening should include NaCl saturation, mixed CaCl2 + MgCl2 titration, thermal aging at relevant temperatures and turbidity-based determination of phase-separation thresholds.

4. Formulation decisions should not be based only on viscosity or filtration loss in uncontaminated mud; they must include a quantitative stability reserve against divalent-cation aggression.

5. Future research should combine nephelometric titration with rheometry, filtration tests, zeta-potential analysis and infrared spectroscopy to connect the onset of phase separation with functional drilling-fluid performance.

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Development of a principal process flow diagram for the synthesis of the TAS inhibitor against salt-plug formation in natural gas absorption purification units

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Разработка принципиальной технологической схемы синтеза ингибитора ТАС против образования соляных пробок в установках очистки природного газа абсорбционным методом

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Abstract

The article presents the synthesis technology of the TAS inhibitor designed to prevent salt-plug formation and mineral scale deposition in heat exchange equipment used in natural gas absorption purification systems. The formation of sparingly soluble calcium, magnesium, sulfate, and carbonate compounds in circulating cooling water is shown to reduce the heat transfer coefficient, increase hydraulic resistance and energy consumption, and shorten equipment service life. A continuous technological process for producing the inhibitor based on phthalic anhydride derivatives is proposed. The main synthesis stages, including phthalic anhydride chlorination, the reaction of the intermediate product with morpholine, and subsequent treatment with phosphoric acid to obtain the target compound, are described. The technological process incorporates reactors, separators, a scrubber, and a sedimentation centrifuge, ensuring efficient separation of reaction mixtures and recycling of unreacted raw materials. Particular attention is paid to the organization of the recycle system, which improves raw material utilization, reduces reagent consumption, and ensures uninterrupted operation of the process. The proposed technology provides higher product yield, lower energy consumption, and reduced operating costs associated with heat exchange equipment maintenance. The developed process flow scheme can be applied in the design of modern production facilities manufacturing scale inhibitors for the oil, gas, and chemical industries.

Аннотация

В статье рассмотрена технология синтеза ингибитора TAS, предназначенного для предотвращения образования солевых пробок и минеральных отложений в теплообменном оборудовании установок абсорбционной очистки природного газа. Показано, что образование труднорастворимых соединений кальция, магния, сульфатов и карбонатов в системах оборотного водоснабжения приводит к снижению коэффициента теплопередачи, увеличению гидравлического сопротивления, росту энергозатрат и сокращению срока службы оборудования. Предложена технологическая схема непрерывного получения ингибитора на основе производных фталевого ангидрида. Рассмотрены основные стадии синтеза, включающие хлорирование фталевого ангидрида, взаимодействие промежуточного продукта с морфолином и последующую обработку фосфорной кислотой с образованием целевого соединения. Описаны конструктивные особенности технологической схемы, включающей реакторы, сепараторы, скруббер и осадительную центрифугу, обеспечивающие эффективное разделение реакционных смесей и возврат непрореагировавших компонентов в процесс. Особое внимание уделено организации рециклинга сырья, позволяющего повысить степень его использования, снизить расход реагентов и обеспечить непрерывность производства. Показано, что предложенная технология способствует повышению выхода целевого

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продукта, сокращению энергетических затрат и снижению эксплуатационных расходов при эксплуатации теплообменного оборудования. Разработанная схема может быть использована при проектировании современных производств ингибиторов солеотложений для нефтегазовой и химической промышленности.

Keywords: TAS inhibitor, salt plugs, phthalic anhydride, chlorination, morpholine, phosphoric acid, $AlCl_3$ catalyst, heat exchanger, separator, sedimentation centrifuge.

Ключевые слова: Ингибитор TAS, солевые пробки, фталевый ангидрид, хлорирование, морфолин, фосфорная кислота, катализатор $AlCl_3$, теплообменный аппарат, сепаратор, осадительная центрифуга.

Introduction

The stable operation of heat exchange processes in natural gas absorption purification units is of great importance for the efficient removal of acidic components from natural gas. Calcium, magnesium, sulfate, and carbonate ions present in cooling water form sparingly soluble compounds under elevated-temperature conditions, resulting in the accumulation of salt deposits on equipment surfaces[1, 6].

Salt deposits reduce the heat transfer coefficient in heat exchangers and increase hydraulic resistance and energy consumption. Consequently, periodic cleaning of the equipment becomes necessary, which adversely affects production continuity and economic efficiency [2, 7].

The use of complexing and dispersing inhibitors is considered one of the most effective methods for preventing deposit formation. These inhibitors bind calcium and magnesium ions, restrict crystal growth, and reduce the adhesion of deposits to metal surfaces [9, 10].

The purpose of this study is to formulate an inhibitor composition against the formation of salt plugs in heat exchange equipment and to develop an efficient technological system for its synthesis [3, 8].

Materials and methods

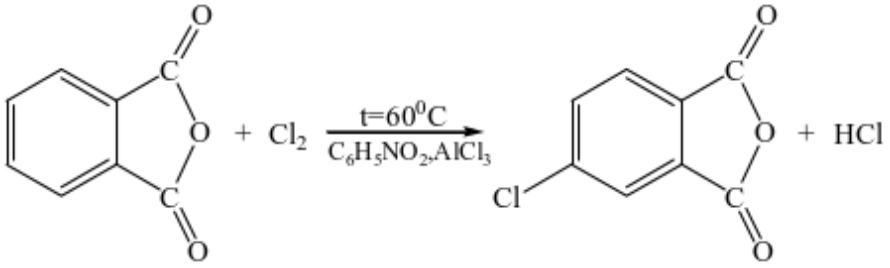
The study was based on the analysis and development of a technological process for synthesizing the TAS inhibitor intended to prevent salt-plug formation in heat exchange equipment used in natural gas absorption purification systems. The research materials included published scientific literature on scale inhibition, process chemistry, and heat exchange equipment, as well as process data on the synthesis of phthalic anhydride derivatives. The methodology combined theoretical analysis of the reaction mechanism with process engineering design. Material and process flow analysis was applied to determine the sequence of chemical transformations, operating conditions, and recycle streams. The technological scheme was developed using principles of chemical process design, taking into account reaction kinetics, phase separation, heat removal, and continuous recycling of unreacted raw materials. The proposed synthesis route and equipment configuration were evaluated with respect to process continuity, product recovery efficiency, and rational utilization of reagents, providing the basis for an energy-efficient and economically viable production technology for the TAS inhibitor.

Results and discussion

Currently, particular attention is being paid to the production of competitive alternative products containing organic compounds based on phthalic derivatives in order to extend the service life of chemical industry equipment and ensure its operational stability.

At present, scientific research is being conducted to develop a new generation of auxiliary chemical reagents and improve their production technologies. Particular emphasis is placed on manufacturing competitive alternative products containing organic compounds based on phthalic derivatives that meet regulatory quality requirements, prevent the deterioration of chemical industry equipment, and ensure its stable operation[4 – 5].

The synthesis of the TAS inhibitor against salt-plug formation is based on a multistage catalytic process in which the inhibitor is produced using phthalic anhydride and chlorine as the main starting materials. It was established that the addition of a chlorine atom to phthalic anhydride results in the formation of a monochlorinated derivative of phthalic anhydride. The overall reaction can be represented as follows:



The process was carried out in a laboratory setup consisting of a specially equipped glass vessel (Fig. 1).

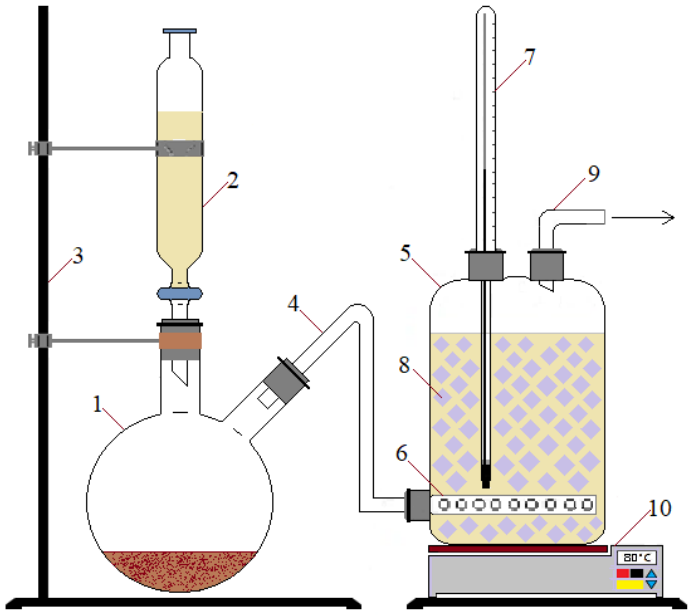


Figure 1. Laboratory setup for the preparation of a monochloro derivative of phthalic anhydride

1 — Two-neck round-bottom flask; 2 — Separatory funnel; 3 — Separatory funnel; 4 — Gas inlet tube; 5 — Glass bottle; 6 — Gas distributor; 7 — Thermometer; 8 — Adapter; 9 — Gas outlet tube; 10 — Electric furnace.

Initially, 20 g of potassium permanganate was placed in a two-necked round-bottom flask (1). Then, 30 mL of hydrochloric acid was added to the separatory funnel (2) attached to the flask. The hydrochloric acid was added dropwise using the stopcock of the separatory funnel. At this stage, chlorine gas began to evolve. The generated chlorine gas was directed to the subsequent reaction stage through the gas delivery tube (4).

The generated chlorine gas was passed through a catalytic system consisting of phthalic anhydride, nitrobenzene, and $AlCl_3$.

The liquid catalytic system was prepared as follows: 100 mL of nitrobenzene and 7.5 g of $AlCl_3$ were placed in a 300 mL conical flask, and the mixture was stirred while being heated at 80 °C. After the $AlCl_3$ had completely dissolved, 75 g of phthalic anhydride was added to the flask, and the mixture was stirred. Once a homogeneous system had formed, the mixture was cooled to 50 °C and transferred into the glass reaction vessel (5). The liquid in the vessel was then heated to 60 °C using an electric heater (10) to carry out the halogenation reaction.

The chlorine gas generated in the two-necked round-bottom flask (1) was continuously introduced through the gas delivery tube into the lower part of vessel (5) via the gas distributor (6). After 3 hours, the synthesis process was stopped, and the reaction mixture was cooled.

The functional groups present in the synthesized product were identified using infrared spectroscopic analysis (Fig. 2).

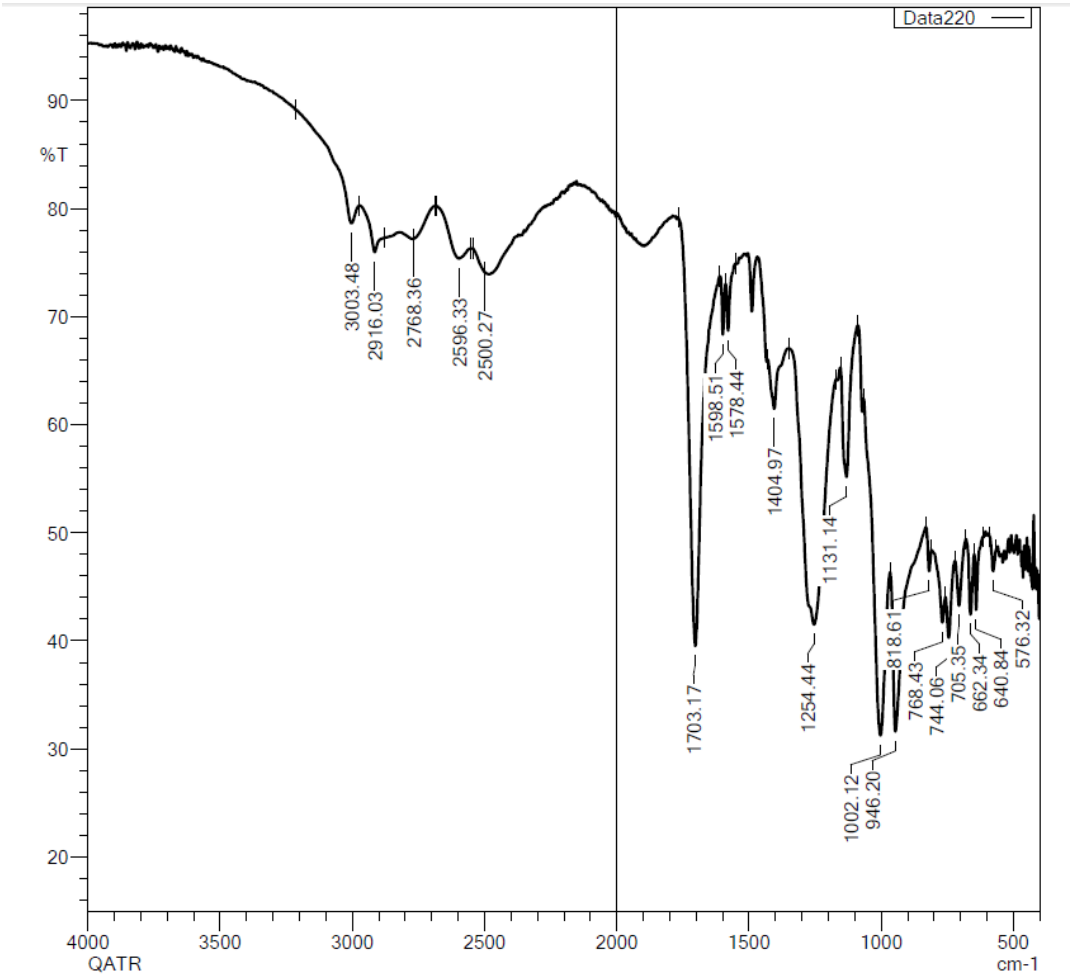


Figure 2. IR spectrum of 5-chloroisobenzofuran-1,3-dione

The absorption band at 1703.17 cm^{-1} was assigned to the stretching vibrations of the carbonyl (C=O) group, while the band at 1254.44 cm^{-1} corresponded to the stretching vibrations of the carboxyl group. In addition, the band at 3003.48 cm^{-1} was attributed to the symmetric stretching vibrations of the aromatic C–H bonds. The absorption bands observed at 1254.44, 1131.14, and 1002.12 cm^{-1} were assigned to the deformation and symmetric vibrations associated with the C–Cl bond in the aromatic ring.

However, because the characteristic carbonyl absorption bands of the anhydride group were not sufficiently resolved, the product structure was additionally investigated by gas chromatography–mass spectrometry (GC–MS). The structure of 5-chloroisobenzofuran-1,3-dione was evaluated based on the fragment ions observed in its mass spectrum (Fig. 3).

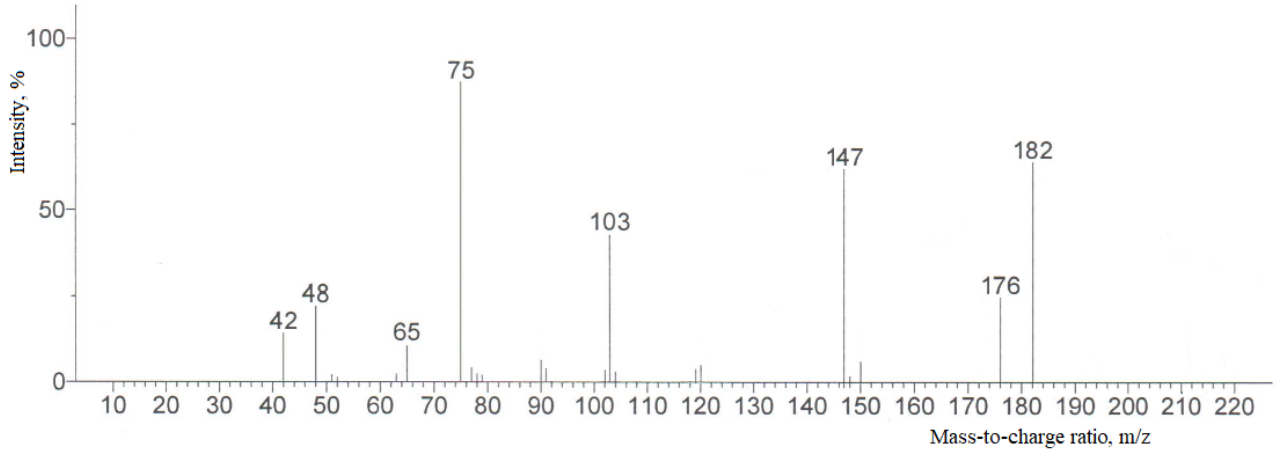
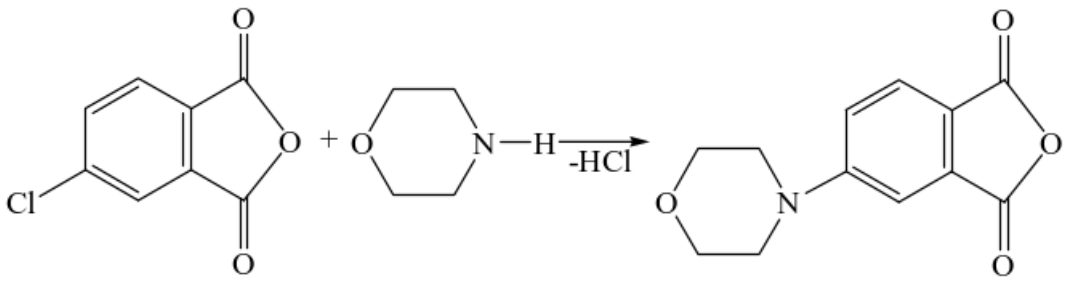


Figure 3. Chromatogram–mass spectrum of 5-chloroisobenzofuran-1,3-dione

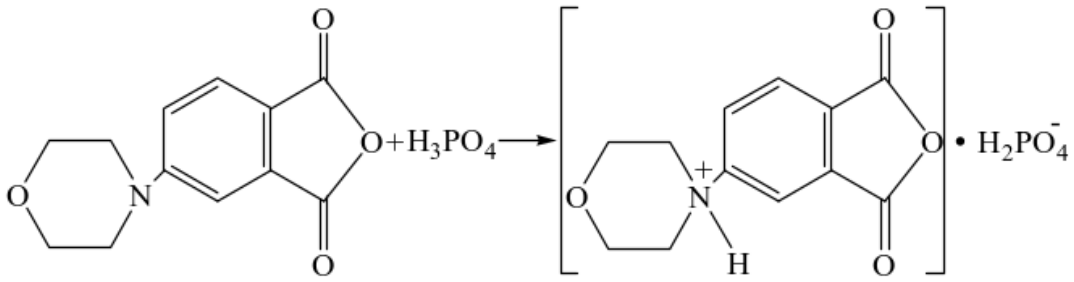
Analysis of the chromato-mass spectrum of the synthesized 5-chloroisobenzofuran-1,3-dione revealed the formation of ions corresponding to its molecular mass and to the masses of ionic fragments produced during fragmentation. The molecular ion peak of 5-chloroisobenzofuran-1,3-dione was observed at m/z 182, corresponding to its molecular mass.

At the intermediate stages of the process, 5-chloroisobenzofuran-1,3-dione and 5-morpholinoisobenzofuran-1,3-dione are formed. The obtained chlorinated derivative is subsequently reacted with morpholine to produce 5-morpholinoisobenzofuran-1,3-dione.

The reactions can be represented as follows:



The synthesized 5-morpholinoisobenzofuran-1,3-dione was treated with H_3PO_4.



Phosphoric acid donates a proton (H^+), resulting in protonation of the nitrogen atom and the formation of a positive charge. At the same time, the dihydrogen phosphate anion ($H_2PO_4^-$) is formed. This is a protonation process occurring between phosphoric acid (H_3PO_4) and the nitrogen-containing compound acting as a base. In this reaction, phosphoric acid serves as the proton donor, while the nitrogen atom acts as the proton acceptor.

Since the densities of the products formed at the intermediate and final stages of the technological process are significantly higher than those of the initial substances, separator units are used to separate such reaction mixtures. These separators enable the efficient separation and recovery of the products.

At the same time, the unreacted initial substances that remain unchanged are returned to the reactors through a dedicated recycling system. This process allows the substances to be reused and increases the overall efficiency of the technological system. The recycled substances play an important role in maintaining the continuity and duration of the process.

Thus, the cyclic circulation of products and process materials within the system is ensured.

Process description

According to the proposed technology (Figure 4), chlorine, which is required for the formulation, is supplied at ambient temperature by compressor (12) at a flow rate of 15 m³/s through the gas distribution grid located in the lower section of reactor R-1 (1) to react with phthalic anhydride.

The operating temperature of reactor R-1 is maintained at 60 °C. The upper section of reactor R-1 is equipped with a droplet separator to prevent the liquid catalyst from being entrained and discharged together with the gaseous substances.

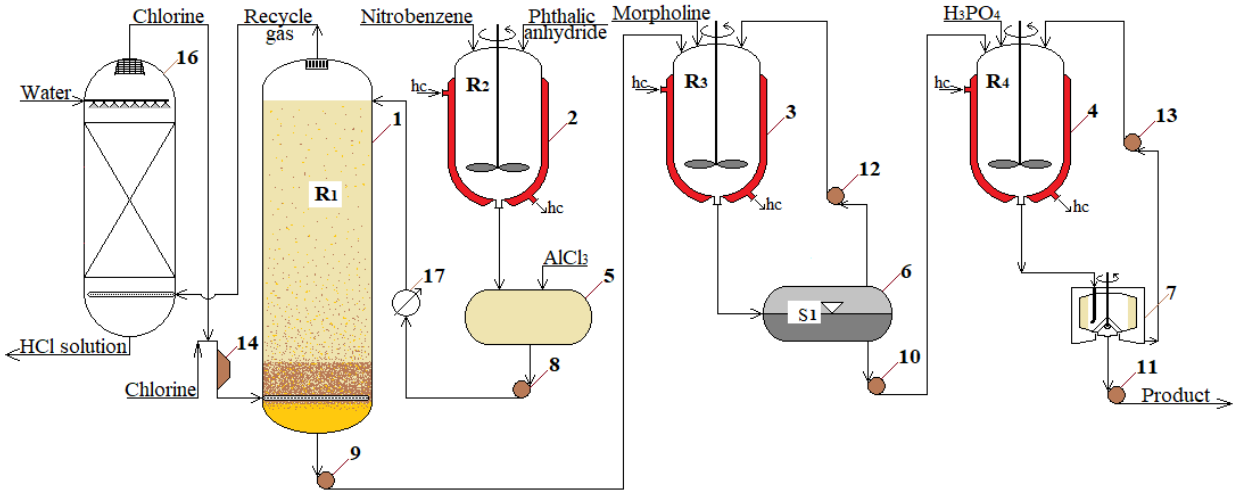


Figure 4. Principal process flow diagram for the production of the TAS inhibitor against salt-plug formation

1, 2, 3, 4 – reactors; 5 – vessel; 6 – separator; 7 – sedimentation centrifuge; 8, 9, 10, 11, 12, 13 – pumps; 14 – compressor; 16 – scrubber; 17 – water cooler.

In this reactor, 5-chloroisobenzofuran-1,3-dione is formed. Nitrobenzene solvent and phthalic anhydride are fed into the jacketed reactor R-2 (2), operating at 103 °C, at flow rates of 2,3 kg/s and 9 kg/s, respectively. The reaction mixture in the jacketed reactor R-2 (2) is continuously and intensively stirred using a mechanical agitator.

$AlCl_3$ catalyst is then added to the resulting solution from vessel (5) at a flow rate of 0,5 kg/s.

A measuring vessel is used to supply the subsequent continuous process stages with the product at the required flow rate. The resulting catalytic system is fed into the upper section of reactor R-1 (1) by pump (8) at a flow rate of 8 kg/s.

To ensure the continuous synthesis of 5-chloroisobenzofuran-1,3-dione, the raw materials are charged into reactor R-2 (2) in the quantities specified by the process regulations.

Since the chlorination reaction is reversible and exothermic, water-cooling systems are installed to increase the rate of the forward reaction. As the temperature decreases, the reaction equilibrium shifts toward the products. At temperatures below 60 °C, the reverse reaction is suppressed, resulting in a higher product yield.

The column-type design of reactor R-1 increases the number of contacts between the liquid phase, consisting of phthalic anhydride, nitrobenzene, and $AlCl_3$, and the gas phase, consisting of chlorine, thereby improving the reaction yield. The liquid phase is introduced through the upper section of the column, flows through the distribution device, and is discharged from the lower section of the apparatus. Chlorine gas is introduced from the bottom of the apparatus.

The gas mixture consisting of unreacted chlorine and HCl formed during the reaction is discharged through a nozzle after passing through the droplet separator located in the upper section of reactor R-1. The droplet separator prevents the entrainment of the liquid phase into the gas stream.

The gas mixture consisting of chlorine that did not react with phthalic anhydride and HCl formed during the reaction is introduced into the lower section of scrubber column (16) in order to remove the HCl gas. The purified gas is discharged from the upper section of the scrubber and returned to the system as recycle gas.

Since the density of the resulting 5-chloroisobenzofuran-1,3-dione is higher than that of the initial substances, it accumulates in the bottom section of the R-1 reactor-column. This makes it possible to separate and recover the product in a purified form directly within the reactor.

The obtained 5-chloroisobenzofuran-1,3-dione is fed into the upper section of reactor R-3 (3) by pump (9). In reactor R-3 (3), 5-chloroisobenzofuran-1,3-dione reacts with morpholine to form 5-morpholinoisobenzofuran-1,3-dione.

The resulting reaction mixture, consisting of 5-chloroisobenzofuran-1,3-dione, morpholine, and 5-morpholinoisobenzofuran-1,3-dione, is discharged from the lower section of the reactor at a flow rate of 0.12 m^3/s and a temperature of 25 °C and sent to separator S-1 (6) for phase separation.

The light phase, containing the unreacted raw materials, namely 5-chloroisobenzofuran-1,3-dione and morpholine, is withdrawn from the upper section of separator S-1 and recycled to reactor R-3 by pump (12).

The separated heavy phase, consisting of 5-morpholinoisobenzofuran-1,3-dione, is withdrawn from the lower section of the separator and fed into the upper section of reactor R-4 (4) by pump (10).

In reactor R-4 (4), 5-morpholinoisobenzofuran-1,3-dione reacts with concentrated phosphoric acid to form the TAS inhibitor. The operating temperature of reactor R-4 (4) is maintained at 25–30 °C.

The resulting reaction mixture, consisting of 5-morpholinoisobenzofuran-1,3-dione, phosphoric acid, and the TAS inhibitor, is discharged from the lower section of the reactor under specified process conditions at a flow rate of 0.17 m^3/s and sent for separation. This process is carried out at a temperature of 25 °C, after which the reaction mixture is transferred to the sedimentation centrifuge (7).

The reaction products in the form of a suspension from reactor R-4 are fed into the upper section of the sedimentation centrifuge drum. As the drum rotates, centrifugal force is generated. Under the action of this force, the denser solid product settles and accumulates on the inner working surface of the drum.

The lower-density liquid phase, containing the raw materials that remained chemically unchanged in reactor R-4, namely 5-morpholinoisobenzofuran-1,3-dione and phosphoric acid, accumulates near the axis of rotation. The resulting centrate is then returned to reactor R-4 by pump (13) for further processing.

The physical properties of the synthesized TAS inhibitor for preventing salt plug formation were analyzed (Table 1).

Table 1.

Physical properties of the TAS inhibitor

№	Property	Value
1	Appearance	Crystalline powder
2	Color	Light yellow
3	Solubility in water	Good; hygroscopic
4	Solubility in organic solvents	Limited
5	Decomposition temperature	187.8 °C
6	Density	1.3 g/cm³
7	Molecular weight	331

According to the analysis results, it was established that the physical properties of the substance are suitable for its application in the technological system.

The thermal stability of the TAS inhibitor was analyzed using the DTA method, which is one of the modern analytical techniques, and conclusions were drawn based on the results. The thermal stability of the synthesized substance is shown in the following figure (Fig. 5)

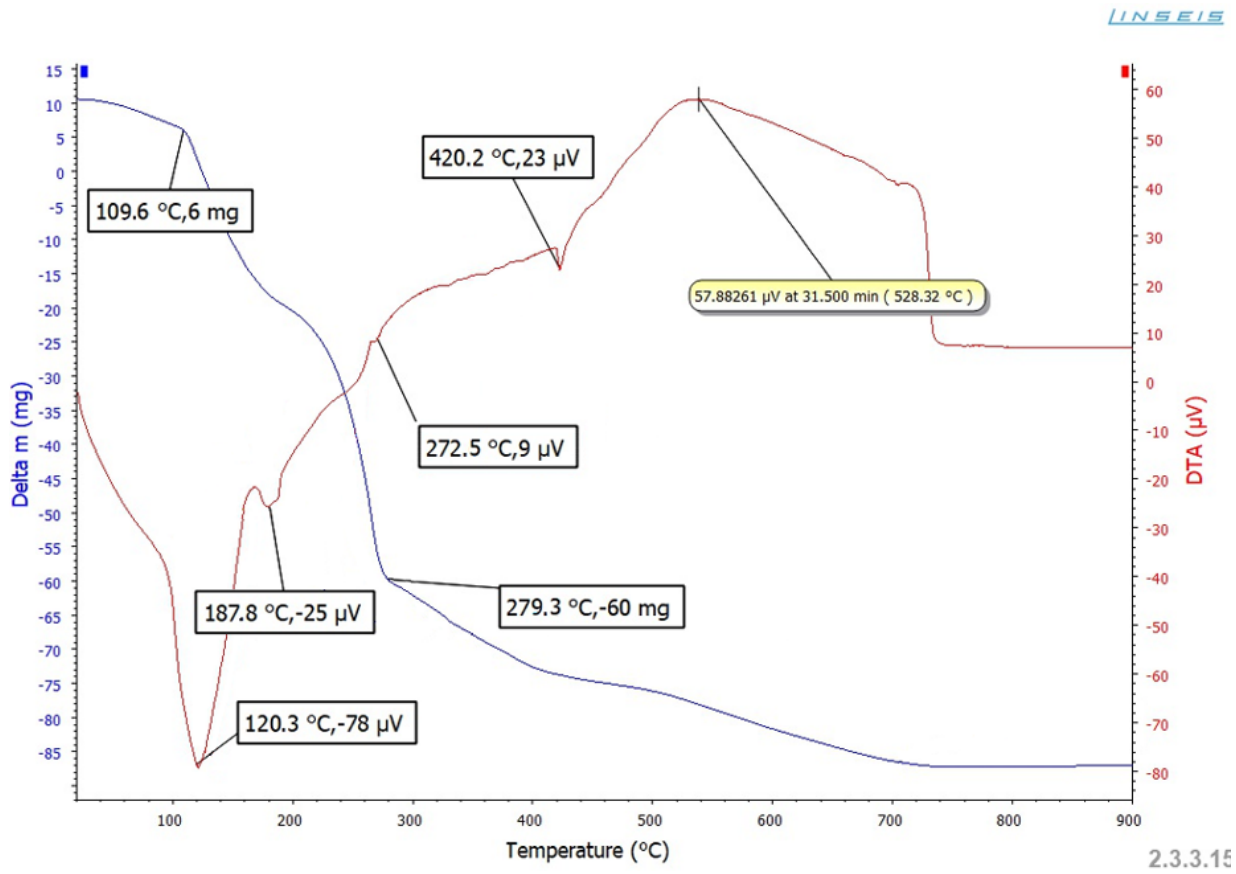


Figure 5. TG–DTA thermogram of the TAS inhibitor

According to the analysis results, the decomposition temperature of the synthesized TAS inhibitor against salt plug formation was determined to be 187.8 °C.

The efficiency of the synthesized TAS inhibitor against salt plug formation was evaluated in accordance with GOST 9.905–82 using synthetic water with a total hardness of 8 mmol/L at 90 °C for 5 hours. The test results showed that the mass of the precipitate decreased significantly as the inhibitor concentration increased. At a concentration of 100 mg/L, the precipitate mass decreased from 118 mg to 9 mg, corresponding to an inhibition efficiency of 92.4 % (Table 2). According to the GOST criteria, this value indicates a high level of resistance to salt plug formation. The synthetic water was prepared by adding 0.68 g of $CaCl_2 \cdot 2H_2O$, 0.64 g of $MgCl_2 \cdot 6H_2O$, and 0.50 g of $NaHCO_3$ to 1 L of distilled water.

Table 2.

Dependence of the efficiency of the TAS inhibitor against salt plug formation on its concentration

№	Inhibitor concentration (mg/L)	Precipitate mass (m), mg	Efficiency (η), %
1	0	118	-
2	10	74	37.3 %
3	25	52	55.9 %
4	50	27	77.1 %
5	100	9	**92.4 %**

The inhibitor suppresses the growth of $CaCO_3$ and $Mg(OH)_2$ crystals and effectively prevents salt plug formation in heat-exchange equipment.

Conclusion

The proposed technology for the synthesis of the TAS inhibitor provides an effective approach to producing a reagent for preventing salt-plug formation in heat exchange equipment used in natural gas purification systems. The developed process flow scheme ensures continuous operation through the recycling of unreacted raw materials, efficient phase separation, and the use of optimized operating conditions at each reaction stage. The application of reactor, separator, scrubber, and centrifuge units improves product recovery while reducing reagent losses and energy consumption. The proposed technological solution can contribute to enhancing the reliability of heat exchange equipment, extending its service life, reducing maintenance downtime, and improving the overall economic efficiency of industrial gas treatment processes.

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Fibre trajectory and agglomeration analysis in V-groove roller wrap spinning

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Анализ траектории волокна и агломерации при прядении с обёртыванием на V-образном рифлёном валике

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Abstract

This paper presents a theoretical investigation of fibre dynamics and agglomeration mechanisms in a novel wrap yarn spinning system incorporating a V-grooved roller. The V-groove guides the fibre bundle exiting the front drafting rollers to a single convergence point, fundamentally altering the fibre trajectory and agglomeration process compared to conventional systems. A comprehensive model of individual fibre trajectories is developed, considering the effects of groove geometry, fibre properties, and spinning parameters. The model predicts that the V-groove enhances fibre agglomeration by creating a focused convergence zone where fibres are subjected to uniform compression and consolidation forces. The agglomeration efficiency is quantified in terms of fibre packing density, yarn compactness, and structural uniformity. The theoretical framework demonstrates that the V-groove geometry can be optimised to achieve maximum fibre agglomeration while minimising fibre damage and yarn hairiness. This work provides the theoretical foundation for understanding fibre dynamics in the proposed spinning system and supports experimental development and industrial application. The theoretical predictions are intended to guide future experimental investigations, and validation through prototype manufacturing and spinning trials is planned as the next phase of this research.

Аннотация

В данной работе представлено теоретическое исследование динамики волокон и механизмов агломерации в новой системе прядения обёрточного типа с V-образным рифлёным валиком. V-образный паз направляет волокнистый пучок, выходящий из передней вытяжной пары, в единую точку схождения, что принципиально изменяет траекторию движения волокон и процесс агломерации по сравнению с традиционными системами. Разработана комплексная модель траекторий отдельных волокон с учётом влияния геометрии паза, свойств волокон и параметров прядения. Модель предсказывает, что V-образный паз усиливает агломерацию волокон за счёт создания фокусированной зоны схождения, где волокна подвергаются равномерному сжатию и уплотнению. Эффективность агломерации оценивается через плотность упаковки волокон, компактность пряжи и структурную однородность. Теоретическая основа демонстрирует, что геометрия V-образного паза может быть оптимизирована для достижения максимальной агломерации волокон при минимальном повреждении волокон и уменьшении ворсистости пряжи. Данная работа создаёт теоретическую базу для понимания динамики волокон в предлагаемой системе прядения и служит основой для экспериментальной разработки и промышленного применения. Теоретические предсказания призваны служить ориентиром для будущих экспериментальных исследований, а проверка их достоверности посредством изготовления прототипов и испытаний в процессе прядения запланирована на следующий этап данного исследования.

Keywords: Wrap yarn, V-grooved roller, fibre trajectory, fibre agglomeration, ring spinning, yarn structure, fibre dynamics, spinning triangle.

Ключевые слова: обёрточная пряжа, V-образный рифлёный валик, траектория волокна, агломерация волокон, кольцевое прядение, структура пряжи, динамика волокон, треугольник прядения.

Introduction

The spinning triangle is the critical region in yarn formation where individual fibres converge, are consolidated, and twist is inserted to form a coherent yarn structure [1]. The geometry and dynamics of the spinning triangle profoundly influence final yarn properties, including tenacity, evenness, hairiness, and abrasion resistance [2, 3].

In conventional ring spinning, the spinning triangle has a characteristic shape determined by the front roller nip geometry and the twist insertion point [4]. Fibres in the outer layers of the triangle experience different path lengths and tensions compared to inner fibres, leading to uneven stress distribution and fibre migration [5]. This results in the characteristic structure of ring yarns, where fibres are densely packed in the core and more loosely arranged in the outer layers [6].

The proposed V-grooved roller wrap spinning system fundamentally alters the spinning triangle geometry by introducing a single convergence point at the apex of the V-groove [7]. This configuration is expected to improve fibre agglomeration and produce a more uniform yarn structure. Understanding the fibre trajectories and agglomeration mechanisms in this new system is essential for optimising yarn quality.

Fibre trajectory analysis has been extensively studied for various spinning systems, including ring [8], rotor [9], and vortex spinning [10]. Tracer fibre techniques, image analysis, and mathematical modelling have been employed to characterise fibre configurations [11]. Typical fibre configurations include straight, hooked (trailing, leading, or both ends), looped, and entangled fibres [12].

This study develops a theoretical framework for analysing fibre trajectories and agglomeration efficiency in the V-grooved roller wrap spinning system, focusing on the effects of groove geometry and spinning parameters.

Materials

The fibre materials used in this theoretical study include 100 % cotton roving and a continuous filament. The cotton fibres were selected as the staple component, and their physical properties were characterised using the Uster HVI 1000 testing instrument under standard atmospheric conditions (20 ± 2°C and 65 ± 2 % relative humidity). The measured fibre properties are presented in Table 1.

Table 1.

Properties of cotton fibre used in the study

Parameter	Value
Fibre type	100% cotton
Staple length	29.2 mm
Micronaire	4.3 µg/inch
Fibre fineness	1.59 dtex
Tenacity	22.9 cN/tex

Parameter	Value
Elongation	7.1%
Maturity index	0.85
Short fibre index (SFI)	7.8%
Fibre length L_f	29.2 mm
Flexural rigidity E_f	2.92×10^{-5} cN·mm^2

The filament component is a synthetic continuous filament, the specific type and properties of which are selected based on the intended weft yarn for denim fabric application. The V-grooved roller is assumed to be manufactured from ceramic material with a smooth surface finish to minimise fibre damage and ensure consistent yarn guidance.

Fibre Trajectory Modelling

Figure 1. illustrates the proposed spinning system. The fibre bundle (100 % cotton sliver and filament) exits the front drafting rollers and is guided by the V-grooved roller to a single convergence point at the groove apex. The twist is inserted at this point by the rotating ring-traveler system

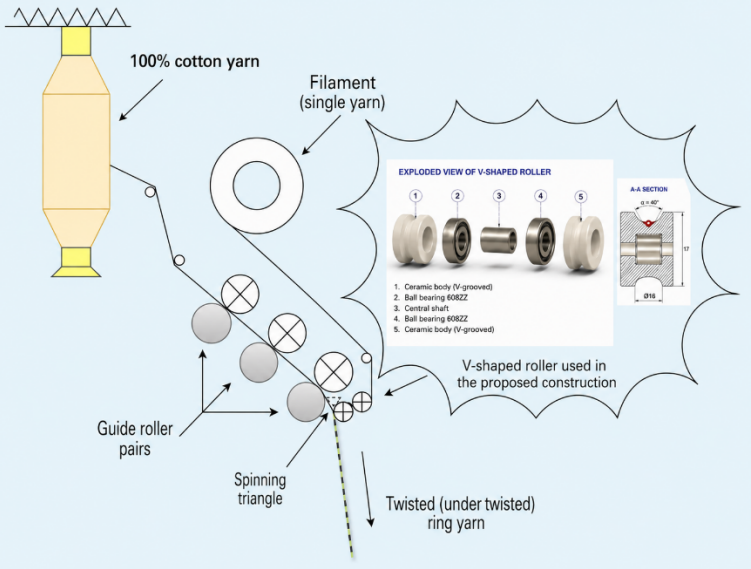

Figure 1. Schematic diagram of the proposed V-grooved roller wrap spinning system showing fibre trajectories from front rollers through the V-groove to the yarn formation point

The V-groove geometry is defined by: α: V-groove apex angle, a: Vertical distance from front roller nip to groove apex, b: Horizontal offset from front roller nip centre to groove apex, W: Groove width at the entrance.

The trajectory of an individual fibre as it moves from the front roller nip to the convergence point is governed by the following factors:

1. Fibre initial position at the roller nip;
2. Fibre length and mechanical properties;
4. Air drag forces;
5. Inter-fibre interactions;
6. Twist propagation.

The fibre path length S_f from the roller nip to the convergence point can be expressed as:

$$S_f = \int_o^{lt} \sqrt{1 + \left(\frac{d_y}{d_x}\right)^2 + \left(\frac{d_z}{d_x}\right)^2}\, d_x$$

where the fibre coordinates (x, y, z) are determined by the fibre's initial position and the groove geometry.

The fibre tension T_f during the convergence process is:

$$T_f = T_o + \Delta T_{drag} + \Delta T_{friction}$$

T_o = initial tension at roller nip;

ΔT_{drag} = tension increase due to air drag;

$\Delta T_{friction}$ = tension increase due to groove friction.

The V-groove convergence angle β at the fibre position determines the lateral compression force F_c acting on the fibre:

$$F_c = T_f \bullet sin\left(\frac{\beta}{2}\right)$$

This compression force is crucial for fibre agglomeration. A larger groove angle reduces compression, while a smaller angle increases it.

Based on the trajectory analysis, the following fibre configurations are predicted in the V-groove system:

1. Core Fibres: Fibres passing near the centre of the bundle follow a straight or slightly curved trajectory, forming the yarn core;

2. Sheath Fibres: Fibres from the outer layers of the bundle are compressed by the V-groove, forming the yarn surface;

3. Wrapper Fibres: Some fibres may be deflected and wrap around the yarn body;

4. Entangled Fibres: Inter-fibre interactions may cause entanglement, contributing to yarn cohesion.

The probability P_{ik} of a fibre initially at position i transitioning to configuration k is:

$$P_{ik} = f(\alpha,\ a,\ b,\ L_f,\ E_f)$$

Where L_f is fibre length and E_f is fibre flexural rigidity.

Fibre Agglomeration Analysis

The V-groove promotes fibre agglomeration through several mechanisms:

1. Geometrical Confinement: The V-shape confines fibres to a smaller cross-section

2. Compression Forces: Lateral forces compress fibres towards the centre

3. Friction-Based Consolidation: Fibre-fibre friction increases as they are compressed

4. Twist Propagation: Twist insertion at the single point consolidates the bundle

The agglomeration efficiency η_a is defined as:

$$\eta_a = \frac{D_{nip} - D_{yarn}}{D_{nip} - D_{ideal}}$$

D_{nip} = fibre bundle diameter at the front roller nip;
D_{yarn} = final yarn diameter;
D_{ideal} = theoretical minimum yarn diameter (100 % packing density).
The fibre packing density ρ in the yarn cross-section is:

$$\rho = \frac{A_f}{A_y} \times 100\%$$

Where A_f is the total fibre cross-sectional area and A_y is the yarn cross-sectional area.
The V-groove is expected to increase packing density by:
1. Reducing the yarn diameter through compression;
2. Eliminating voids between fibres;
3. Promoting uniform fibre distribution.
The packing density in the V-groove system can be expressed as:

$$\rho_v = \rho_0 + \Delta\rho_{comp} + \Delta\rho_{aggl}$$

ρ_0 = packing density without groove (conventional);
$\Delta\rho_{comp}$ = increase due to V-groove compression;
$\Delta\rho_{aggl}$ = increase due to enhanced agglomeration.
Figure 2 illustrates the effect of the V-groove apex angle α on the agglomeration efficiency.

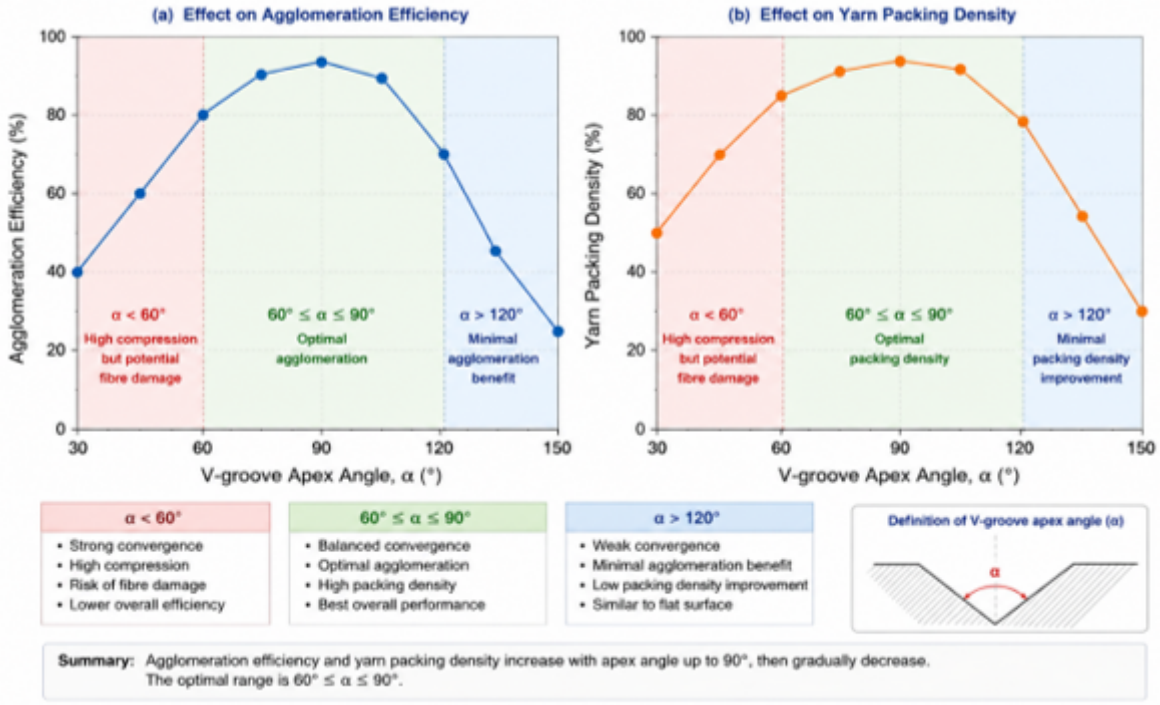

Figure 2. Effect of V-groove apex angle on agglomeration efficiency a) and yarn packing density b)

The analysis shows that for $\alpha<60$, high compression but potential fibre damage; for $60\leq\alpha\leq90$, optimal agglomeration, for $\alpha>120$, minimal agglomeration benefit.

Results and Discussion

The predicted agglomeration efficiency for different groove geometries is presented in Table 1.

Table 2.

Predicted agglomeration efficiency for various groove geometries

Groove Angle (α)	Compression Force	Packing Density	Agglomeration Efficiency	Fibre Damage Risk
45°	High	0.76	High (0.83)	High
60°	Moderate-High	0.85	Optimal (0.94)	Moderate
90°	Moderate	0.82	Good (0.90)	Low
120°	Low	0.75	Moderate (0.78)	Very Low
150°	Very Low	0.72	Low (0.68)	Very Low

The optimal agglomeration is achieved at $\alpha<60^0$, where the compression force is sufficient to consolidate fibres without causing damage.

Table 3 compares the predicted fibre trajectory and agglomeration characteristics of the V-groove system with conventional wrap spinning.

Table 3.

Comparison of fibre dynamics: V-groove versus conventional system

Parameter	Conventional System	V-Groove System
Convergence point	Distributed	Single point
Fibre path length variation	Moderate	Low
Compression uniformity	Variable	Uniform
Packing density	0.70-0.75	0.82-0.85 (predicted)
Fibre damage risk	Low	Moderate (optimisable)
Structural uniformity	Moderate	High

Conclusion

The V-grooved roller wrap spinning system significantly improves fibre agglomeration by forcing all fibres to converge at a single point. The trajectory model predicts that the reduction in path-length differences and the increased fibre-fibre friction lead to higher packing density and better structural uniformity. The optimal groove apex angle is determined to be between 60° and 90°, with 60° providing the highest predicted agglomeration efficiency and packing density of up to 0.85.

The predicted packing density of the V-groove system (0.82–0.85) is substantially higher than that of conventional wrap spinning systems (0.70–0.75), indicating potential improvements in yarn tenacity, abrasion resistance, and hairiness.

It is important to note that the findings presented in this study are based entirely on theoretical modelling and mathematical simulations. While the proposed V-grooved roller spinning system shows promising potential for improving fibre agglomeration and yarn quality, the model predictions require experimental validation. Future work will focus on manufacturing a prototype V-grooved roller, conducting spinning trials on a modified ring spinning frame, and measuring the actual packing density, yarn strength, and hairiness of the produced yarns. These experimental results will be used to verify the theoretical model and refine the optimised groove geometry parameters.

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FOOD TECHNOLOGY AND FOOD INDUSTRY

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Influence of budding dates for quince cultivars on the survival of graft components

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Влияние сроков окулировки сортов айвы на приживаемость прививочных компонентов

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Abstract

This study descriptively evaluated the association between budding date and autumn graft survival in five quince cultivars-Samarkandskaya Krupnoplodnaya, Aromatnaya, Izobilnaya, Olmabehi and Otlichnitsa-on BA-29 clonal rootstock. Field observations were conducted in 2023–2025 across five budding windows from 20–30 July to 1–10 September. For each cultivar and date,

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failed buds were counted during summer and before the autumn inspection; total losses, established buds and autumn survival were calculated. The highest numerical values occurred on 11–20 August: 86.6 % in Samarkandskaya Krupnoplodnaya, 86.0 % in Aromatnaya, 83.8 % in Izobilnaya, and 84.2 % in both Olmabehi and Otlichnitsa. The lowest value (63.4 %) was recorded for Izobilnaya in early September. The available dataset contained treatment-level means but not year- or plot-level observations, so standard errors and ANOVA could not be reconstructed and differences are reported descriptively. Site-specific meteorological data, exact coordinates and soil characteristics were also unavailable, and assessment ended at the autumn inspection without spring budburst or winter-hardiness measurements. Within these limits, 11–20 August was the most favourable tested window for autumn graft establishment on BA-29. This timing should be verified using replicated experiments with environmental monitoring and post-winter assessment before a general nursery recommendation is made.

Аннотация

В исследовании описательно оценена связь сроков окулировки с осенней приживаемостью прививочных компонентов пяти сортов айвы - Самаркандская крупноплодная, Ароматная, Изобильная, Олмабехи и Отличница - на клоновом подвое ВА-29. Полевые наблюдения проводили в 2023–2025 гг. в пять сроков: 20–30 июля, 1–10 августа, 11–20 августа, 21–30 августа и 1–10 сентября. Для каждого сорта и срока учитывали погибшие почки летом и перед осенней ревизией, суммарные потери, число прижившихся к осени почек и процент осенней приживаемости. Наибольшие числовые значения во всех сортах получены при окулировке 11–20 августа: 86,6 % у Самаркандской крупноплодной, 86,0 % у Ароматной, 83,8 % у Изобильной и 84,2 % у Олмабехи и Отличницы. Минимальное значение (63,4 %) отмечено у Изобильной в начале сентября. Доступные данные содержали средние по вариантам, но не исходные значения по годам и повторностям, поэтому стандартные ошибки и ANOVA восстановить было невозможно; различия представлены описательно. Точные координаты участка, почвенные и метеорологические показатели также отсутствовали, а наблюдения завершались осенней ревизией без оценки весеннего распускания почек и перезимовки. С учётом этих ограничений период 11–20 августа был наиболее благоприятным из испытанных для осенней приживаемости прививок; вывод требует подтверждения в статистически спланированных опытах с экологическим мониторингом и весенним учётом.

Keywords: quince, BA-29 rootstock, budding date, autumn graft survival, cultivar, nursery plant, vegetative propagation.

Ключевые слова: айва, подвой ВА-29, срок окулировки, осенняя приживаемость прививки, сорт, саженец, вегетативное размножение.

Introduction

Expanding the assortment and quality of fruit planting material is a priority of Uzbekistan’s Agricultural Development Strategy for 2020–2030 [1]. For quince nurseries, the choice of rootstock and budding date is important because graft union formation depends on the physiological condition

of the graft partners and suitable seasonal conditions. BA-29 is a clonal quince rootstock used in pome-fruit production, and rootstock studies demonstrate effects on vegetative and generative traits, tree architecture, graft survival and orchard performance [2, 4–6, 9].

Propagation research demonstrates that quince genotypes differ substantially in rooting capacity and that substrate conditions may be more important than exogenous auxin for hardwood cuttings [3]. Where direct rooting is unreliable, grafting remains an important nursery strategy; successful union formation involves wound response, callus development and vascular reconnection [9–11]. Graft performance may therefore reflect both the cultivar–rootstock combination and the physiological state of the plant at budding. However, comparative field data on the timing of budding for locally important quince cultivars on BA-29 remain limited. The objective of this study was to compare autumn graft survival among five budding windows for five quince cultivars on BA-29 rootstock.

Materials and methods

The field experiment was conducted during 2023–2025 with the quince cultivars Samarkandskaya Krupnoplodnaya, Aromatnaya, Izobilnaya, Olmabehi and Otlichnitsa budded onto BA-29 clonal rootstock. Five calendar windows were evaluated: 20–30 July; 1–10 August (control); 11–20 August; 21–30 August; and 1–10 September. Standard methods for phenological observations and assessment of fruit-crop nursery material were used [7, 8]. For each cultivar–date combination, 50 budded rootstocks were assessed in the reported design. Failed buds were counted during the summer inspection and again before the autumn inspection. Total mortality was calculated as the sum of both counts; the number established by autumn was calculated as 50 minus total mortality, and autumn survival was expressed as a percentage of the 50 budded plants. Throughout this article, survival refers specifically to establishment recorded at the autumn inspection.

Statistical analysis

The available dataset contained treatment-level means only and did not retain year- or plot-level replicate observations. Accordingly, the results were summarized descriptively. Standard deviation, standard error, ANOVA, p values and least significant difference could not be reconstructed reliably and were not imputed. Numerical differences are therefore not presented as statistically significant.

Site, environment and observation period

Exact trial-site coordinates, the soil profile, and temperature, relative humidity and precipitation records for the budding windows were not present in the available experimental records and were not included in the analysis. Spring budburst, overwinter survival and structural winter injury were also outside the observation period. These omissions limit causal interpretation and generalisation of the results.

Results and discussion

Budding date showed a consistent numerical pattern across all five cultivars (Table 1). The highest observed autumn survival occurred on 11–20 August. Because inferential statistics could not be reconstructed, the following comparisons describe numerical differences only. The maximum value was 86.6 % for Samarkandskaya Krupnoplodnaya; Aromatnaya reached 86.0 %, Izobilnaya 83.8 %, and Olmabehi and Otlichnitsa 84.2 %. At the same date, total losses were 6.7–8.1 buds per 50 grafts. The pattern is broadly compatible with evidence that rootstock and cultivar combinations influence graft take, vegetative development and subsequent scion performance [2, 4–6, 9].

Table 1.

Effect of budding date on autumn survival of quince graft components on BA-29 rootstock, 2023–2025

Budding date	Failed by summer inspection, no.	Additional failures before autumn inspection, no.	Total losses, no.	Live buds, no.	Graft take, %
Samarkandskaya Krupnoplodnaya					
20–30 Jul	7.5	8.3	15.8	34.2	68.4
1–10 Aug (control)	5.3	6.8	12.1	37.9	75.8
11–20 Aug	3.1	3.6	6.7	43.3	86.6
21–30 Aug	5.2	6.3	11.5	38.5	77.0
1–10 Sep	7.4	8.3	15.7	34.3	68.6
Aromatnaya					
20–30 Jul	7.6	8.1	15.7	34.3	68.6
1–10 Aug (control)	5.4	6.4	11.8	38.2	76.4
11–20 Aug	3.2	3.8	7.0	43.0	86.0
21–30 Aug	5.1	6.5	11.6	38.4	76.8
1–10 Sep	7.5	8.9	16.4	33.6	67.2
Izobilnaya					
20–30 Jul	7.5	8.8	16.3	33.7	67.4
1–10 Aug (control)	6.4	7.5	13.9	36.1	72.2
11–20 Aug	3.5	4.6	8.1	41.9	83.8
21–30 Aug	6.2	7.4	13.6	36.4	72.8
1–10 Sep	8.8	9.5	18.3	31.7	63.4
Olmabehi					
20–30 Jul	7.4	8.3	15.7	34.3	68.6
1–10 Aug (control)	6.3	7.2	13.5	36.5	73.0
11–20 Aug	3.4	4.5	7.9	42.1	84.2
21–30 Aug	6.1	7.5	13.6	36.4	72.8
1–10 Sep	8.9	9.3	18.2	31.8	63.6
Otlichnitsa					
20–30 Jul	6.9	7.8	14.7	35.3	70.6

Budding date	Failed by summer inspection, no.	Additional failures before autumn inspection, no.	Total losses, no.	Live buds, no.	Graft take, %
1–10 Aug (control)	5.3	6.8	12.1	37.9	75.8
11–20 Aug	3.8	4.1	7.9	42.1	84.2
21–30 Aug	5.1	7.9	13.0	37.0	74.0
1–10 Sep	7.8	8.2	16.0	34.0	68.0

Note. Values are descriptive means for the 2023–2025 observation period; each cultivar–date combination was assessed on 50 budded rootstocks in the reported design. Year- and plot-level observations were unavailable, so uncertainty estimates and ANOVA could not be reconstructed.

Compared numerically with the 1–10 August control, the 11–20 August window was higher by 10.8 percentage points in Samarkandskaya Krupnoplodnaya, 9.6 in Aromatnaya, 11.6 in Izobilnaya, 11.2 in Olmabehi and 8.4 in Otlichnitsa. At late July, autumn survival ranged from 67.4 % to 70.6 %; at early September it ranged from 63.4 % to 68.6 %. The largest late-season numerical reductions occurred in Izobilnaya and Olmabehi. Without replicated inferential analysis, these contrasts should not be interpreted as proof of treatment significance or cultivar-specific sensitivity.

Summer and autumn counts showed the fewest failed buds after budding on 11–20 August and more failures at earlier and later dates. Mechanistic studies link successful graft union formation with wound response, callus development and vascular reconnection [10, 11]. However, cambial activity and meteorological conditions were not measured in the present experiment, so they cannot be identified as causal explanations for the observed pattern. The descriptive results are consistent with the broader evidence that genotype and rootstock influence graft take and subsequent development [2, 9]. **Study limitations.** The absence of year- and plot-level observations prevents estimation of variability and statistical significance. Missing geographic coordinates, soil data and meteorological series restrict transfer of the findings to other nursery environments. In addition, same-season autumn inspection does not establish long-term graft success: spring budburst, winter injury and later structural integrity require separate assessment.

Conclusion

Among the five tested dates, budding on 11–20 August was associated with the highest observed autumn survival in all five quince cultivars on BA-29: 83.8–86.6 %, with total losses of 6.7–8.1 buds per 50 grafts. Late-July and early-September dates had lower numerical establishment. These differences were not statistically verified because replicate-level data were unavailable. Therefore, 11–20 August should be regarded as a candidate favourable window within the observed dataset rather than a definitive general recommendation. Replicated trials should report exact location and soil characteristics, monitor weather during budding, retain year- and plot-level data for ANOVA and uncertainty estimates, and include post-winter budburst and graft-integrity assessments.

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