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Investigation of the process for producing nitrogen-phosphorus fertilizers by processing Shorsu phosphorites with wet-process phosphoric acid activated with ammonium nitrate

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Исследование процесса получения азотно-фосфорных удобрений путем переработки фосфоритов Шорсу экстракционной фосфорной кислотой, активированной аммиачной селитрой

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Abstract

This study investigates the production of complex nitrogen-phosphorus fertilizers containing calcium and magnesium from low-grade phosphate rock from the Shorsu deposit (Fergana Region, Uzbekistan), containing 6,87 % P_2O_5 . To enhance the efficiency of raw material decomposition, wet-process phosphoric acid (WPA) containing 19,50 % P_2O_5 , produced from washed calcined phosphate concentrate of the Central Kyzylkum deposits and activated with ammonium nitrate (NH_4NO_3), was used. The effects of the phosphorite-to-WPA mass ratios (100:80 and 100:100), neutralization of the slurry with gaseous ammonia to pH 3,1 – 3,5, and drying at 100 – 105°C on the product composition and the degree of conversion of phosphates into plant-available forms [K(avail.)] were investigated. The optimal process conditions were determined as follows: phosphorite-to-WPA (100 % P_2O_5 basis) ratio of 100:100, temperature of 80°C, and process duration of 90 – 120 min. Under these conditions, the resulting concentrated complex fertilizer contained 43,63 % total P_2O_5 , 42,15 % available P_2O_5 , 2,85 % N, 6,46 % CaO, and 4,56 % MgO, while the relative content of available phosphates with respect to total phosphates [K(avail.)] reached 96,61 %. The results demonstrate the feasibility of efficiently utilizing low-grade Shorsu phosphorites for the production of multicomponent fertilizers containing nitrogen, phosphorus, calcium, and magnesium.

Аннотация

В данной работе исследован процесс получения сложных азотно-фосфорных удобрений, содержащих кальций и магний, из низкосортного фосфоритного сырья месторождения Шорсу (Ферганская область, Узбекистан) с содержанием P_2O_5 6,87 %. Для

повышения эффективности разложения сырья использовалась экстракционная фосфорная кислота (19,50 % P_2O_5), полученная на основе мытого обожженного фосфоконцентрата Центральных Кызылкумов и активированная нитратом аммония (NH_4NO_3). В исследовании изучено влияние массовых соотношений фосфорит: ЭФК (100:80 и 100:100), нейтрализации пульпы газообразным аммиаком до $pH=3,1 - 3,5$ и сушки при температуре 100 – 105°C на состав продукта и степень перехода фосфатов в усвояемую форму $[K(усв.)]$. Определены оптимальные условия процесса: соотношение фосфорит:ЭФК (100 % P_2O_5) — 100:100, температура – 80°C, продолжительность – 90 – 120 минут. При этом в составе полученного концентрированного сложного удобрения содержание P_2O_5 общ. составило 43,63 %, P_2O_5 усв. – 42,15 %, N – 2,85 %, CaO – 6,46 % и MgO – 4,56 %, а относительное содержание усвояемой формы фосфатов к их общей форме $[K(усв.)]$ достигло 96,61 %. Результаты демонстрируют возможность эффективного использования бедных фосфоритов Шорсу для производства многокомпонентных удобрений, содержащих азот, фосфор, кальций и магний.

Keywords: phosphorite, low-grade phosphate rock, extraction phosphoric acid, ammonium nitrate, activation, ammoniation, retrogradation, nitrogen-phosphorus fertilizer, calcium- and magnesium-containing fertilizer.

Ключевые слова: фосфорит, низкосортное фосфатное сырье, экстракционная фосфорная кислота, нитрат аммония, активация, аммонизация, ретроградация, азотно-фосфорное удобрение, кальций- и магниесодержащее удобрение.

Introduction

Worldwide, including in the Republic of Uzbekistan, the demand for complex fertilizers containing not only the primary macronutrients (N, P, and K) but also secondary nutrients such as calcium and magnesium is steadily increasing. Such multicomponent fertilizers are of particular importance for increasing crop yields and improving soil fertility [10; 11; 17]. At the same time, due to the annual depletion of conventional high-grade phosphate resources, there is growing interest in utilizing relatively poor, compositionally complex, and low-grade phosphate ores for the production of phosphorus-containing fertilizers [12; 14-16].

The industrial production of wet-process phosphoric acid (WPA) from low-grade phosphate rocks containing approximately 20 % P_2O_5 is economically inefficient due to the high consumption of reagents, the large amount of impurities, and the need for additional processing costs. Consequently, such phosphate raw materials are used only to a limited extent, mainly for the production of single superphosphate. However, conventional single superphosphate produced from low-grade phosphate rocks is characterized by a relatively low nutrient content (typically about 9 – 11 % P_2O_5 and 1 – 2 % N). This reduces the efficiency of fertilizer production and application and also increases the relative costs associated with its transportation, packaging, and storage [18].

The partial neutralization of wet-process phosphoric acid obtained from phosphate rocks with a high content of magnesium and other foreign impurities negatively affects product quality. When secondary phosphate is processed with such acid, its decomposition rate does not exceed 50%, resulting in a fertilizer that fails to meet standard requirements. To increase this indicator, it is

necessary to introduce 2–3 times more acid into the system compared to the stoichiometric norm, or to employ methods for its pre-activation. However, an increase in the acid norm raises the amount of free acid in the finished product, deteriorating its physical and mechanical properties. To eliminate this problem and improve product quality, a technology is proposed that involves neutralizing the acidic slurry formed during the decomposition stage with gaseous ammonia, followed by granulation and drying [17; 18].

To increase the reactivity of wet-process phosphoric acid used in the processing of natural phosphates, various chemical additives are introduced into its composition. Strong mineral acids, such as nitric and sulfuric acids, or salts, such as ammonium nitrate and ammonium sulfate, can be used as such activating components. Research has investigated in detail the processes of decomposing low-grade phosphorites with a low phosphorus pentoxide (P_2O_5) content (13 % or more) using WPA activated through these methods, as well as obtaining high-quality phosphorus fertilizers from them [10; 11].

Phosphate rocks with an extremely poor chemical composition (less than 13 % P_2O) also occur in nature. Due to such a low content of phosphorus pentoxide and the high amount of foreign impurities in the raw material, the processes of their direct chemical processing into mineral fertilizers have not been sufficiently studied to date. This determines the necessity of the complex utilization of the local raw material base and the development of new technological solutions for activating such low-grade ores.

Significant reserves of extremely low-grade phosphate ore are concentrated in the Shorsu region of the Uzbekistan District, Fergana Region, Uzbekistan. The P_2O_5 content of these ores is only 2 – 8 %, which substantially limits their direct use in conventional phosphorus fertilizer production technologies. The phosphate rock investigated in the present study contains 6,87 % P_2O_5 , along with significant amounts of calcium and magnesium compounds. Therefore, the development of an efficient processing method capable of converting this low-grade local phosphate raw material into a concentrated multicomponent fertilizer is of both scientific and practical importance.

The present study investigates a technological approach based on the decomposition of Shorsu phosphorite using wet-process phosphoric acid produced from washed calcined phosphate concentrate (WCPC) of the Central Kyzylkum deposits and pre-activated with ammonium nitrate (NH_4NO_3).

The processes of WPA activation in the presence of ammonium nitrate have been studied in detail previously. The WPA used in the present study contained 19,50 % P_2O_5 , and ammonium nitrate was added at an amount corresponding to 1,5 % of the acid mass for its activation. WPA activated with ammonium nitrate is used to intensify the decomposition of relatively low-grade phosphate raw materials. Subsequent ammoniation and drying of the acidic phosphate slurry obtained by decomposing the phosphate raw material with the activated acid are employed to optimize the chemical composition of the product and increase its nitrogen content while maintaining a high proportion of plant-available phosphates.

Accordingly, the aim of this study is to determine the technological conditions for processing extremely low-grade Shorsu phosphorite using WPA activated with ammonium nitrate and to evaluate the effects of the phosphorite-to-WPA ratio, ammoniation, and phosphate slurry drying on the degree of conversion of phosphates into plant-available forms and on the chemical composition of the resulting complex fertilizer containing calcium and magnesium.

Materials and Methods

Raw materials and their preparation. Low-grade phosphate ore from the Shorsu deposit, located in the Uzbekistan District of the Fergana Region, Uzbekistan, was used as a secondary phosphate raw material. Its chemical composition was as follows (wt.%): $P_2O_5 = 6,87$; $CaO = 19,60$; $MgO = 4,20$; $CO_2 = 13,63$; $K_2O = 1,97$; $Fe_2O_3 = 0,48$; $FeO = 2,24$; $Al_2O_3 = 0,37$; $SO_3 = 0,52$, and other components. To increase the specific contact surface area of the raw material during acid decomposition, the phosphorite was ground and sieved to obtain a fraction with a particle size of less than 0,25 mm.

Wet-process phosphoric acid (WPA), produced industrially by the dihydrate process from washed calcined phosphate concentrate (WCPC) of the Central Kyzylkum deposits, was used for the decomposition of the secondary phosphate raw material. The WPA had the following chemical composition (wt.%): $P_2O_5 = 19,50$; $CaO = 0,28$; $MgO = 0,85$; $Fe_2O_3 = 0,57$; $Al_2O_3 = 0,63$; $F = 1,18$; $SO_3 = 2,67$. Prior to phosphorite decomposition, the WPA was chemically activated. A commercial-grade sample of dry ammonium nitrate (NH_4NO_3) was used as the activating additive.

Experimental procedure. The production of a complex nitrogen-phosphorus fertilizer containing the macronutrients calcium and magnesium was carried out under laboratory conditions using a method analogous to the continuous process for double superphosphate production. The experimental procedure comprised the stages of acid activation, phosphorite decomposition, ammoniation of the phosphate slurry, evaporation of the ammoniated slurry, followed by its drying and granulation by mixing with recycle material.

Activation of WPA. A predetermined amount of NH_4NO_3 was added to the initial WPA, after which the mixture was continuously stirred until a homogeneous solution was obtained.

Acid decomposition of phosphorite. A three-necked flask was placed in a water bath maintained at $80^\circ C$. A stirrer equipped with a liquid seal was inserted through the central neck of the flask and driven by an electric motor. A water-cooled reflux condenser was fitted into the second neck. A predetermined amount of WPA was introduced through the third neck using a dropping funnel [at a phosphorite:WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:80 – 100, corresponding to 100 – 120 % of the stoichiometric amount required for the formation of monocalcium phosphate from phosphorite]. A predetermined amount of ammonium nitrate was then added through the funnel, and the mixture was stirred until the salt was completely dissolved. Subsequently, a pre-weighed amount of the secondary phosphate raw material – Shorsu phosphorite – was introduced through the same neck. After the addition of phosphorite was completed, a thermometer was inserted into the same neck of the flask. The phosphate slurry was stirred for 90 – 120 min, with the reaction time measured from the beginning of phosphorite addition to the activated WPA.

Ammoniation of the phosphate slurry. To enrich the product with nitrogen, the acidic superphosphate slurry obtained after decomposition of the secondary phosphate raw material with activated WPA was neutralized with gaseous ammonia (NH_3) to a pH of 3,1-3,5.

Evaporation, drying, and granulation of the ammoniated slurry. The resulting ammoniated slurry was evaporated until the residual moisture content reached 20 – 25 %. One portion of the evaporated slurry was dried at 100 – 105°C for use as recycle material. It was then mixed with the remaining portion of the slurry until the moisture content of the resulting mass reached 8 – 10 %. The resulting mass was granulated and dried in a drying oven at 100 – 105°C. The granules of the final product were sieved, and the commercial-size fraction of 2 – 4 mm was collected. The oversized fraction (>4 mm) was crushed and mixed with the powder fraction (<1 mm) generated during sieving; this fine material was returned to the process as recycle material.

Chemical analysis and calculation of the degree of conversion of phosphates into plant-available [K(avail.)] and water-soluble [K(w.s.)] forms. The components present in the products and slurries obtained by decomposition of natural phosphorite with activated WPA (wet-process phosphoric acid) and subsequent ammoniation, namely total, available, and water-soluble P_2O_5 , free P_2O_5 [1, 7, 9], CaO and MgO [3, 9], SO_3 [5, 9, 13], N [6, 8, 9], Fe_2O_3 , Al_2O_3 , F and K_2O [9], moisture (H_2O) [2, 9], as well as the pH of the slurries [4; 9], were determined using analytical methods based on the applicable standards.

The efficiency of phosphate conversion into plant-available and water-soluble forms was evaluated in terms of the degrees of conversion into the available and water-soluble forms [K(avail.) and K(w.s.), respectively], calculated using the following equations:

$$K_{\text{avail.}} = \frac{P_2O_{5\text{avail.}}}{P_2O_{5\text{total}}} \times 100$$

$$K_{\text{w.s.}} = \frac{P_2O_{5\text{w.s.}}}{P_2O_{5\text{total}}} \times 100$$

where: $\text{P}_2\text{O}_{5\text{avail.}}$ and $\text{P}_2\text{O}_{5\text{w.s.}}$ are the contents of plant-available P_2O_5 and water-soluble P_2O_5 in the product, respectively; $\text{P}_2\text{O}_{5\text{total}}$ is the total P_2O_5 content in the fertilizer.

Results and discussion. The technological parameters of the process for producing calcium- and magnesium-containing nitrogen-phosphorus fertilizers using low-grade phosphorite (6,87 % P_2O_5), as well as the chemical composition of the resulting products, are presented in Tables 1 and 2.

Initially, a comparative analysis was carried out to evaluate the chemical characteristics of the initial acidic slurry obtained after the phosphoric acid decomposition of Shorsu phosphorite and the intermediate ammoniated slurry formed during its subsequent neutralization with gaseous ammonia.

As shown in Table 1, when activated WPA containing 19,50 % P_2O_5 was used at a phosphorite-to-WPA mass ratio of 100:80, after decomposition for 1,5 – 2,0 h (90 – 120 min), the degree of conversion of phosphates into the plant-available form [K(avail.)] reached 95,90 %.

Table 1.

Technological parameters of Shorsu phosphorite decomposition with activated wet-process phosphoric acid and the chemical composition of the resulting phosphate slurry

Parameters and components	Chemical composition of the slurry components (wt.%) and technological process parameters			
	Acidic slurry		Ammoniated slurry	
Mass ratio of phosphorite to WPA (calculated on a 100% P_2O_5 basis)	100:80	100:100	100:80	100:100
WPA rate relative to the stoichiometric requirement, %	100	125	100	125
Slurry pH	3,2	2,4	3,5	3,1
P_2O_5 (total)	19,26	19,81	19,98	20,86
P_2O_5 (available)	18,47	19,33	18,46	20,12
P_2O_5 (water-soluble)	14,44	16,75	13,77	17,09
K_2O (total)	0,17	0,19	0,18	0,20
CaO (total)	3,82	3,17	3,76	3,09
MgO (total)	2,31	2,28	2,26	2,19
N (total)	0,43	0,45	0,57	1,35
H_2O	54,46	56,11	52,76	53,68
$(P_2O_5\text{avail.}/P_2O_5\text{total}) \cdot 100$ [K(avail.)], %	95,90	97,57	93,89	96,45
$(P_2O_5\text{w.s.}/P_2O_5\text{total}) \cdot 100$ [K(w.s.)], %	74,97	84,38	68,92	81,93

During the neutralization with gaseous ammonia of the slurry obtained at a phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:80, reverse chemical transformations associated with phosphorus retrogradation (conversion of phosphates into plant-unavailable forms) occurred to a certain extent. As the slurry medium changed with increasing pH, the readily plant-available mono- and dicalcium phosphates were partially converted into sparingly soluble tricalcium phosphate, resulting in a decrease in the proportion of plant-available phosphates. During ammoniation of the acidic slurry, the pH increased from 3,2 to 3,5, while the P_2O_5 content increased from 19,26 to 19,98 %. As a result of the retrogradation of 2,01 % of the plant-available phosphates, K(avail.) decreased from 95,90 to 93,89 %. At the same time, the proportion of water-soluble phosphates [K(w.s.)] also decreased markedly from 74,97 to 68,92 %.

Increasing the WPA rate had a positive effect on the degree of phosphorite decomposition. When the acid rate was increased to 1,25 times the stoichiometric requirement, i.e., when the phosphorite-to-WPA mass ratio was increased to 100:100, K(avail.) in the acidic slurry increased to 97,57 %, while K(w.s.) increased to 84,38 %. During neutralization of the acidic slurry with gaseous ammonia from an initial pH of 2,4 to a final pH of 3,1, despite the observed increases in total P_2O_5 from 19,81 to 20,86 %, plant-available P_2O_5 from 19,33 to 20,12 %, and water-soluble P_2O_5 from 16,75 to 17,09 %, the ratio of plant-available phosphates to total phosphates [K(avail.)] decreased from 97,57 to 96,45 %, while the proportion of water-soluble phosphates [K(w.s.)] decreased from 84,38 to 81,93 %.

The slurries obtained during the decomposition of natural phosphorite with activated WPA contained approximately 52 – 56 % water. Subsequently, the slurry was evaporated to reduce its water content to 20 – 25 %. After the addition of dry fertilizer as recycle material, the moisture content was further reduced to 8 – 10 %, after which the resulting mass was granulated and dried at 100 – 105°C.

During the drying of the acidic and ammoniated slurries obtained by decomposition of Shorsu phosphorites with activated WPA, the chemical equilibrium in the system underwent further changes. The experimental results obtained for the dried products are presented in detail in Table 2.

Table 2.

Chemical composition of acidic and ammoniated products obtained from Shorsu phosphorite and activated WPA and the technological process parameters

Parameters and components	Chemical composition of the product components (wt.%) and technological process parameters			
	Acidic product		Ammoniated product	
Mass ratio of phosphorite to WPA (calculated on a 100% P ₂ O ₅ basis)	100:80	100:100	100:80	100:100
WPA rate relative to the stoichiometric requirement, %	100	125	100	125
Slurry pH	3,2	2,4	3,5	3,1
P ₂ O ₅ (total)	41,40	44,50	41,46	43,63
P ₂ O ₅ (available)	39,76	43,84	38,94	42,15
P ₂ O ₅ (water-soluble)	28,15	35,81	25,70	34,24
P ₂ O ₅ (free)	1,76	8,42	0,41	3,57
K ₂ O (total)	0,36	0,43	0,38	0,41
CaO (total)	8,24	7,12	7,80	6,46
MgO (total)	5,02	5,10	4,69	4,56
N (total)	0,92	1,03	1,18	2,85
H ₂ O	2,21	1,37	1,95	3,22
(P ₂ O ₅ avail./P ₂ O ₅ total)·100 [K(avail.)], %	96,04	98,52	93,92	96,61
(P ₂ O ₅ w.s./P ₂ O ₅ total)·100 [K(w.s.)], %	68,00	80,47	61,99	78,48

During the drying of phosphate slurries, further decomposition of the residual phosphorite present in the slurry may continue. As a result, depending on the amount of free acid remaining in the slurry, the proportion of plant-available P₂O₅ may increase slightly. When the acidic and ammoniated slurries obtained at a phosphorite-to-WPA ratio of 100:80 were dried at 100 – 105°C, the K(avail.) values of the resulting products reached 96,04 % and 93,92 %, respectively. Compared with the corresponding values for the initial slurries, these increases were only 0,14 % and 0,03 %, respectively. The contents of free P₂O₅ in the resulting acidic and ammoniated products were 1,76 % and 0,41 %, respectively.

Although the degree of conversion of phosphates into the plant-available form [K(avail.)] reached 93,92 % after ammoniation and subsequent drying of the slurry obtained by decomposition of natural phosphorite with activated WPA at a phosphorite-to-WPA mass ratio (calculated on a

100 % P_2O_5 basis) of 100:80, this value was below the established quality requirements. Therefore, the production of an ammoniated fertilizer under these conditions was considered technologically impractical.

In contrast, when the slurry obtained at the same phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:80 was dried without prior ammoniation, the K(avail.) value of the resulting product reached 96,04 %. In addition, the free P_2O_5 content of the product (1,76 %) met the established technological requirements. Therefore, under these conditions, direct drying of the acidic slurry provided a higher proportion of plant-available phosphates than neutralization followed by drying.

Thus, the acidic product obtained by decomposing Shorsu phosphorite with WPA activated with ammonium nitrate at a phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:80, followed by slurry evaporation, granulation, and drying, had the following chemical composition (wt.%): total P_2O_5 = 41,40; plant-available P_2O_5 = 39,76; water-soluble P_2O_5 = 28,15; free P_2O_5 = 1,76; K_2O = 0,36; CaO = 8,24; MgO = 5,02; N = 0,92; H_2O = 2,21, along with other components, and met the established technological requirements. The proportion of plant-available phosphates in the product, $(P_2O_5\text{avail.}/P_2O_5\text{total}) \times 100$, was 96,04 %, while the proportion of water-soluble phosphates, $(P_2O_5\text{w.s.}/P_2O_5\text{total}) \times 100$, was 68,0 %.

When the amount of activated WPA used for the decomposition of natural phosphorite was increased to 1,25 times the stoichiometric requirement [at a phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:100], the K(avail.) value of the resulting product increased by 2,48 percentage points, reaching 98,52 %. Nevertheless, the free P_2O_5 content of the final product was 8,42 %, which exceeded the established limit and therefore restricted the applicability of this product.

In this case, i.e., at a phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:100, the most favorable results were achieved by neutralizing the resulting acidic slurry with gaseous ammonia. After ammoniation, the K(avail.) value of the slurry was 96,45 %, and during subsequent drying it increased slightly to 96,61 %. At the same time, the free P_2O_5 content of the final product decreased to 3,57 %. The calcium- and magnesium-containing nitrogen-phosphorus fertilizer obtained under these optimal conditions demonstrated the most favorable combination of high phosphate availability and reduced residual free acidity.

Conclusion

The feasibility of producing a high-quality concentrated complex fertilizer using low-grade Shorsu phosphorite containing only 6,87 % P_2O_5 and WPA containing 19,50 % P_2O_5 , activated with ammonium nitrate, was demonstrated.

It was established that at a phosphorite-to-activated WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:80, the ammoniated product was characterized by a relatively low degree of phosphate conversion into the plant-available form [K(avail.) = 93,92 %], indicating that the production of an ammoniated fertilizer under these conditions is technologically impractical. However, drying the acidic slurry without prior ammoniation resulted in a product with a K(avail.) value of 96,04 %.

The following optimal conditions were determined for producing a calcium- and magnesium-containing nitrogen-phosphorus fertilizer by decomposing Shorsu phosphorite with pre-activated WPA, followed by neutralization with gaseous ammonia, evaporation, drying, and granulation of the slurry: initial activated WPA concentration of approximately 19 % P_2O_5 ; WPA rate for phosphorite decomposition of 125 % of the stoichiometric requirement [corresponding to a phosphorite-to-WPA mass ratio (calculated on a 100 % P_2O_5 basis) of 100:100]; decomposition temperature of 80°C; process duration of 90 – 120 min; neutralization of the slurry with gaseous ammonia to pH 3,1 – 3,5; and drying and granulation of the slurry at 100 – 105°C.

The fertilizer obtained under these conditions had the following chemical composition (wt. %): total P_2O_5 = 43,63; plant-available P_2O_5 = 42,15; water-soluble P_2O_5 = 34,24; free P_2O_5 = 3,57; K_2O = 0,41; CaO = 6,46; MgO = 4,56; N = 2,85; and H_2O = 3,22. The proportion of plant-available phosphates in the product, $(P_2O_{5\text{avail.}}/P_2O_{5\text{total}}) \times 100$, reached 96,61 %, while the proportion of water-soluble phosphates, $(P_2O_{5\text{w.s.}}/P_2O_{5\text{total}}) \times 100$, was 78,48 %.

Thus, the proposed processing method provides an effective approach for converting extremely low-grade Shorsu phosphorite into a concentrated multicomponent fertilizer containing phosphorus, nitrogen, calcium, and magnesium.

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