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CATALYTIC PROCESSING OF NATURAL BITUMEN FROM BITUMINOUS SANDS

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Abstract: *Introduction.* Bituminous sands deposits are a very rich source of energy in bitumen. However, access to the main part of these reserves is difficult, and the conversion of bitumen into liquid, low-sulfur products require innovative solutions, since the properties of traditional oil and bitumen are significantly different. *Methods.* To obtain the necessary products from bituminous sands, it is first necessary to separate organic part from the bituminous sands, and only then it can be further processed. Currently, industrial bitumen production in Canada is carried out using mining technology. In this regard, Kazakhstan needs to develop domestic technology in accordance with its West Kazakhstan conditions and domestic demand. *Results and discussion.* High-temperature cracking processes of West Kazakhstan natural bitumens in the presence of mesoporous aluminosilicate, synthetic zeolite and natural zeolite catalysts have been studied. The catalysts lead to the formation of liquid products up to 92.2%, reducing the number of tar-asphaltene compounds from 52.4% to 16.3%. *Conclusion.* Natural bitumen from the Beke and Munaily Mola fields is classified as heavy oil and consists mainly n-alkanes, terpanes, and steranes. A mesoporous aluminosilicate catalyst enabled efficient catalytic cracking, giving up to 89% liquid products and increasing oil and light fraction yields. High activity is attributed to the large pore size. Cracking destroys aliphatic and polynaphthenic structures in asphaltenes, increases aromaticity, and reduces oxygen-containing compounds, while resins show increased aromaticity due to the breakdown of naphthenic rings and alkyl groups.

Key words: natural bitumen, catalysts, cracking, tar-asphaltene compounds, reaction direction

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1. Introduction

Kazakhstan's oil sector occupies a leading position in the national economy. Today, many of Kazakhstan's oil and gas fields were discovered in the 1930s and 1940s and are in the late stages of development. Most of these fields are hard-to-recover, highly viscous oils, and over 40% are sulfur and high-sulfur. Therefore, the oil and gas industry will have to increasingly focus on developing and bringing heavy, highly viscous, hard-to-recover oil fields into commercial production [1].

Direct cracking of heavy feedstocks is the simplest method technically. However, the presence of impurities in the feedstock deactivates the catalyst, promotes intensive coke formation, reduces process selectivity, and degrades the quality of the products. Pre-treatment of residual feedstock increases product yield and quality, but increases the cost of the process. Solvent-based catalytic cracking is most often used to upgrade feedstock. Deasphalting using various hydrocarbon solvents from C_3 to C_5 or their mixtures, as well as light gasolines. Less common are thermal adsorption deasphalting processes using contacts with large pores, a small surface area, and low activity. The deasphalted products and distillates obtained from these processes can be used (directly or after desulfurization) as feedstock for catalytic cracking units. These processes result in the removal of resins and asphaltenes, fairly deep demetallization , and partial desulfurization and denitrogenation, which significantly facilitates subsequent processing by catalytic processes [2-5].

The conversion of heavy crude oils into synthetic crude [6] involves mild thermal cracking of fuel oil in soaking chambers downstream of the atmospheric column. The possibility of additionally introducing physical energy into the visbreaking process in the form of acoustic cavitation is indicated. The developed technology enables the production of up to 70% synthetic crude, with a 20% increase in gasoline fractions and a 17% increase in diesel fractions compared to the original crude, and approximately 30% residue. The residual product after the thermal polycondensation unit has a density of 990-1000 kg/m^3 and, in quality, corresponds to the most liquid grades of road bitumen.

A variant of processing natural bitumen, called Donor Refined Bitumen [7], where the distillate fractions are first distilled from the bitumen, and the residue with a boiling point above 500°C is mixed with a hydrogen donor solvent. The latter is the circulating product of the process, which is pre- hydrogenated to restore the donor capacity. Hydrocracking of the mixture is carried out at a temperature of 410-460°C and a pressure of 3.5-5.5 MPa, with the degree of conversion reaching 70%. By mixing the final product with light fractions of bitumen, synthetic oil is obtained, which does not contain residual fractions. However, the use of hydrocracking as the main process for processing natural bitumen requires higher hydrogen consumption and the involvement of significant resources of natural gas or other hydrocarbon feedstock for its production.

Currently, global experience is based on approximately 20 processes for upgrading natural bitumen, deposits of which are at various stages of development [8-10]. However, if we consider projects that have reached the industrial level, it is necessary to note that technologies traditionally used for processing heavy residues are being employed: coking, hydrogenation, and deasphalting. There is an ongoing debate regarding the optimal method for increasing the hydrogen:carbon ratio. Many researchers believe that delayed coking offers potential. The advantage of this process is the absence of hydrogen production, which significantly reduces capital investment. However, the low quality of the products in terms of sulfur and unsaturated hydrocarbon content, as well as problems with the use of low-grade coal, are increasingly limiting the use of this technology.

Natural bitumens and heavy oils are characterized by high contents of metals, N, S, and O, which create difficulties during oil refining. Concentrations of heteroatom-containing compounds in oil may be relatively low, but their impact during oil refining can be significant. The presence of these compounds in the catalytic system creates significant process limitations, causing catalytic poisoning and catalyst deactivation [11, 12]. In finished products, heteroatom-containing compounds can also cause problems, including instability during storage and discoloration. Therefore, studying the composition of high-molecular heteroorganic compounds in oil is very important.

A literature review reveals that natural bitumen from oil-bituminous rocks is an important source of hydrocarbon feedstock, and interest in it is growing annually. However, its use as a feedstock requires efficient extraction of bitumen from -bituminous sands, study of the physicochemical composition and properties, and development of processing processes. Extraction of natural bitumen from bituminous sands is dependent on the chemical composition and nature of the natural bitumen, its resin, asphaltene, and heteroatom content. Therefore, important objectives of this study include extracting natural bitumen from bituminous sands and studying the changes in high-molecular compounds during thermal processing of natural bitumen using catalytic additives. Thus, it follows from the above that cracking natural bitumen produces synthetic crude oil whose characteristics (density, viscosity, molecular weight and others) exceed those of conventional crude oils, and its processing using a conventional process flowsheet can yield a wide range of hydrocarbon components.

2. Experimental part

A distinctive feature of Kazakhstan's bituminous deposits is the outcrop of bituminous sands at the surface. In these reactivated deposits, in zones of active water exchange, light fractions have been lost, and the residual petroleum components have been exposed to various supergene factors, which have led to a heavier composition and the formation of slow-moving extra-heavy oils and solid bitumens. Bitumen deposits occur in the form of small lenses and interlayers. Visual analysis of a sample of bituminous sands reveals a rich black color with a bitumen sheen. All rock grains are coated and glued together by viscous bitumen. At a temperature of 20°C, the bituminous sands are difficult to separate into individual pieces. When the temperature rises to 50-60°C, due to the decrease in bitumen viscosity, the sands is easily crushed with a spatula and forms a mastic-like mass. The organic part of the bituminous sands was extracted by solvent. The solvent extraction method allows to determine the exact amount of organic matter in bituminous sands and facilitates the complete extraction of the organic part. Extraction of natural bitumen from bituminous sands was carried out with chloroform.

Table 1 - Physical-chemical characteristics of natural bitumens

Indicator	Bituminous sand deposit	
	Beke	Munaily Mola
Content in bituminous sands, wt. %	10-12	16-25
Density, g/cm^3	0.99	0.99
Total sulfur content, %	1.5	1.4
Ash content, %	0.4	0.5
Coking ability, %	30	35
Viscosity at 80°C, cSt	21	26
Softening temperature, °C	20	41
Pour point, °C	18	16
Content of mechanical impurities, wt. %	0.08	0.11

Table 1 present physical and chemical characteristics of natural bitumens from two bituminous sands. Natural bitumen extracted from the Beke deposit is characterized by the following indicators: density 0.99 g/cm^3; freezing point +18°C; degree of coking 30%; ash content 0.4%; softening point +20°C. The content of natural bitumen in the sands of the Munaily Mola deposit ranges from 16 to 25% and is characterized by high values of density, viscosity, and degree of coking. Extra-heavy oils also have these characteristics.

Methodology for thermal cracking of natural bitumen

Thermal catalytic cracking of natural bitumen was conducted in a steady-state mode in 12-ml autoclave reactors. The bitumen mass loaded into the reactor was 7 g. The experiments were conducted in air, which does not significantly change the composition of the resulting products due to the small volume, at a temperature of 450°C for 60 minutes. During the experiment, the mass of the reactor without a sample and the mass of the reactor with a sample, prepared for the experiment, were recorded. After the heat treatment of natural bitumen, the yield of gaseous products was determined by the mass loss of the reactor with a sample after removing the gas products from the reactor. After collecting the liquid products, the reactor was washed with chloroform and weighed. The resulting difference between the mass of the reactor before and after the experiment was defined as coke.

Table 2 presents the characteristics of the mesoporous aluminosilicate (MP), zeolite in active hydrogen form (HY), as well as natural zeolite (NZ) catalysis. The zeolite catalyst (HY) has a high specific surface area of 874 m^2/g with a small pore size (7.4 nm). In [12], it was shown that this catalyst increases the depth of destruction of the resinous components of heavy oil with the production of low-boiling fractions. The MP catalyst has a lower specific surface area (600 m^2/g), but its pore size is almost 7 times higher and is 50 nm. MP was used based on the assumption that it is the large pore size that will increase the depth of destruction of resins and asphaltenes without developing a coke deposition reaction. Zeolite heulandite-clinoptilolite from the Tayzhuzgen deposit (East Kazakhstan region, Tarbagatai district), activated by acid treatment and calcination, was used as a natural catalyst.

Table 2 - Characteristics of natural bitumen cracking catalysts

Catalyst	Pore size, nm	Specific surface area, m^2/g	Silicate module	Concentration of acid sites, μmol/g
MP	50	600	20	
HY	7.4	874	4.9	929
NZ	10.4	7.2		

Methodology for determining the material composition of liquid products

The organic matter composition was determined using a traditional approach: first, the asphaltene content of the sample was determined using the "cold" Golde method. The resin concentration in the resulting maltenes was then determined by adsorption. The analyzed product was applied to activated silica gel, the mixture was placed in a Soxhlet extractor, and the hydrocarbon components (oils) were sequentially eluted with n-hexane and the resins with an ethanol-benzene mixture (method 1217-2005, Institute of Petroleum Chemistry).

3. Results and discussion

Thermal degradation processes for heavy hydrocarbon feedstocks can increase the yield of low-boiling liquid products, forming coke and gas as byproducts. Cracking processes in the presence of various catalysts are of particular interest. Thermal cracking of natural bitumen produces liquid products and coke, along with a small amount of gas (Table 3). The yield of liquid cracking products from Munaily Mola bitumen was 6% higher than from Beke bitumen, while the yield of coke was lower 4.7%. Thermal processing increases the oil content, while the total amount of high-molecular-weight components in the bitumen decreases. This is likely due to increased coke formation and the breakdown of resinous components, resulting in lighter products. The data obtained indicate that the high-molecular-weight components of Munaily Mola bitumen are more readily cracked than those of Beke bitumen.

The most probable mechanism of catalytic cracking of hydrocarbons is considered to be the carbonium ion mechanism:

Catalyst + Bitumen → Coke/Catalyst + Liquid Product + Gas

Coke/catalyst + $2O_2 \rightarrow$ Catalyst + $2CO + CO_2$ (regeneration)

Table 3 - Material balance of the process of thermal catalytic cracking of bitumen

Process	Content, wt . %		
	Gas	Coke	Liquid products
Natural bitumen from the Beke deposit			
Natural bitumen	0	0	100.0
Cracking	1.4	30.9	67.7
Cracking with mesoporous cat.	2.2	8.8	89.0
Cracking with synthetic zeolite	2.7	7.7	89.6
Cracking with natural zeolite	1.0	19.1	79.9
Natural bitumen from the Munaily Mola deposit			
Original bitumen	0	0	100.0
Cracking	0.2	26.2	73.6
Cracking with mesoporous cat.	1.4	7.4	91.2
Cracking with synthetic zeolite	1.6	6.2	92.2
Cracking with natural zeolite	1.2	13.4	85.4

Catalytic cracking of bitumen with a mesoporous catalyst reduces coke yield by 3.5 times for both types of bitumen. For Beke bitumen, the yield of gaseous products increases from 1.4% to 2.2%, while for Munaily Mola bitumen, the gas content increases sevenfold to 1.4% by weight. Oil content increases by more than 1.5 times compared to the initial content. This increase is due to the extensive degradation of resins, the amount of which in the cracking products decreases by more than fourfold.

The use of a synthetic zeolite (HY) catalyst also significantly slows down coke formation, with the yield of solid cracking products being 1% lower by weight compared to bitumen cracking with an MP catalyst. This catalyst allows for the significant destruction of high-molecular components of bitumen (more than 51% relative), but their content in liquid cracking products is higher than in experiments using an MP catalyst.

A natural zeolite catalyst is a catalytic additive for thermal cracking and hydrogenolysis of heavy hydrocarbons. Natural zeolite prevents secondary polymerization of the resulting low-molecular-weight fragments, is resistant to sulfur poisoning, and is capable of binding nitrogen and sulfur from the products. It is also inert to the mineral component of bituminous sands and the metals present in bitumen. At the same time, the coke and gas yields of both fields decreased by 12.2% and 11.6%, respectively, at the Beke and Munaily Mola fields, compared to thermal cracking.

Table 4 - Group composition of products of thermal catalytic cracking of natural bitumen

Process	Content, wt. %		
	Oils	Resins	Asphaltenes
Natural bitumen from the Beke deposit			
Natural bitumen	49.2	44.9	5.9
Cracking	61.3	28.3	10.4
Cracking with mesoporous cat.	77.2	13.7	9.1
Cracking with synthetic zeolite	72.8	16.8	10.4
Cracking with natural zeolite	77.0	14.4	8.6
Natural bitumen from the Munayily Mola deposit			
Natural bitumen	47.6	46.4	6.0
Cracking	83.6	13.4	3.0
Cracking with mesoporous cat.	79.7	11.1	9.2
Cracking with synthetic zeolite	75.8	13.2	11.0
Cracking with natural zeolite	61.5	27.3	11.2

Table 4 presents the results of an analysis of the component composition of the products of cracking of natural bitumen. The natural zeolite catalyst affected the material composition of natural bitumen from the Beke field, with the oil content reaching 77% and the resin content decreasing by 14.4%. For natural bitumen from the Munaily Mola field, the yield of high-molecular components was 22.1% higher than with cracking. The data obtained show that the total content of resinous-asphaltene components in the cracking products of Munaily Mola bitumen changes (compared to the composition of the initial bitumen), with the asphaltene content decreasing by 50% and the resin content by 29%. However, when processing Munaily Mola bitumen with a mesoporous catalysis, despite a significant increase in the yield of liquid cracking products and their oil content, an increase in the asphaltene component content is observed. Moreover, the oil content in the liquid cracking products of Munaily Mola bitumen is 2% higher than that of the Beke bitumen. Cracking with synthetic zeolite led to increasing the resin and asphaltene content 22% for Munaily Mola bitumen and 27% for Beke bitumen. The resin content with the natural zeolite decreased by 30% for Beke bitumen compared to the composition of the initial bitumen, and the asphaltene content increased by 2%. Munaily Mola bitumen led to increasing the oil content 14% compared to the composition of the initial bitumen.

Thus, it has been established that the use of catalysts for these objects in the cracking process increases the destruction of high-molecular-weight components (up to 50% relative). The use of mesoporous and zeolite catalysts significantly enhances the cracking process. The presence of a mesoporous structure in the synthesized aluminosilicate ensures the accessibility of its active sites located in the bulk to large molecules of petroleum feedstock (resins and asphaltenes), where they undergo destruction. Metal particles promote disproportionation and redistribution of hydrogen from high-molecular-weight compounds to components of gasoline and diesel fractions, thereby inhibiting coke formation.

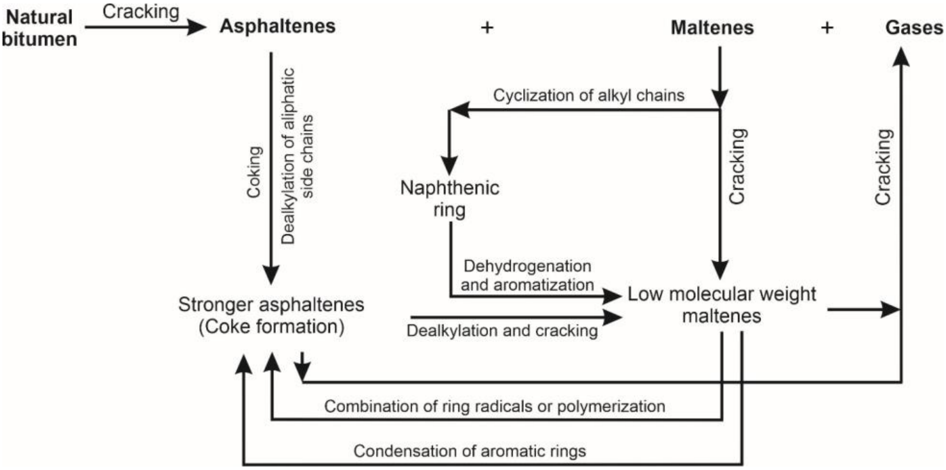

Figure 1 - Scheme of reactions of catalytic cracking of natural bitumen

Cracking at 450°C causes chemical structural changes, resulting in the formation of asphaltenes, coke, maltenes, and a small amount of gas (Figure 1). Asphaltenes in bitumen form coke, resins, and gaseous products. Coke formation occurs through dealkylation of the long aliphatic side chains of asphaltenes. The resulting strong asphaltenes, in the presence of a catalyst, form low-molecular-weight maltenes. Gaseous cracking products are then formed from the low-molecular-weight maltenes and more stable asphaltenes. Maltenes are converted at high temperatures via two reaction pathways: alkyl chain cyclization and cracking. Alkyl chain cyclization leads to the formation of an intermediate product a naphthenic ring. Naphthenic hydrocarbons then form more stable products through aromatization and dehydrogenation. As the concentration of low-molecular-weight maltenes in the system increases, the following reactions occur: radical combination (polymerization) and condensation of aromatic rings. All these reactions lead to the formation of more stable asphaltenes and then coke. Thus, thermal processing of bitumen involves primary and secondary reactions. Primary reactions during thermal cracking of bitumen mainly proceed in three directions: stable asphaltenes (coke), maltenes, and gases. Strong asphaltenes and maltenes can also undergo secondary reactions, such as dealkylation of aliphatic side chains, cyclization of alkyl chains, recombination of ring radicals or polymerization, dehydrogenation and aromatization of naphthenic rings, and condensation of aromatic rings.

4. Conclusion

The group and detailed physical-chemical composition of natural bitumen from the Beke and Munayly Mola fields shows that both hydrocarbons are heavy oils and the composition of the oils is represented by n-C_{22}-C_{25} -alkanes, aromatic hydrocarbons such as terpanes (pentacyclanes), steranes (tetracyclanes).

A mesoporous aluminosilicate catalyst was proposed for the catalytic cracking of natural bitumen from the Beke and Munaily Mola deposits. This catalyst resulted in liquid products with an 89% yield and increased oil content (77%) and light fractions. The high activity of the mesoporous catalyst is due to the large pore size (50 nm).

As a result of cracking, aliphatic and polynaphthenic structures in the asphaltene structure are destroyed, aromaticity increases, and the number of oxygen-containing structures decreases. In the resin structure, the aromaticity factor increases with the destruction of naphthenic rings and alkyl substituents.

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МҰНАЙБИТУМДЫ ЖЫНЫСТАРДАҒЫ ТАБИҒИ БИТУМДАРДЫ КАТАЛИТИКАЛЫҚ ӨҢДЕУ

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Түйіндеме. *Кіріспе.* Мұнайбитумды жыныстар кенорындары битумға өте бай энергия көзі болып табылады. Дегенмен, бұл қорлардың негізгі бөлігіне қол жеткізу қиын, ал битумды сұйық, аз күкіртті өнімдерге айналдыру инновациялық шешімдерді қажет етеді, себебі дәстүрлі мұнай мен битумның қасиеттері айтарлықтай ерекшеленеді. *Әдістер.* Мұнайбитумды жыныстан қажетті өнімдерді алу үшін алдымен битумды жыныстан оны бөліп алу керек, содан кейін ғана оны әрі қарай өңдеуге болады. Қазіргі уақытта Канадада өнеркәсіптік битум өндірісі тау-кен технологиясын қолдану арқылы жүзеге асырылады. Осыған орай, Қазақстан өзінің Батыс Қазақстан жағдайына және ішкі сұранысқа сай отандық технологияны дамыту керек. *Нәтижелер және талқылау.* Батыс Қазақстан табиғи битумдарының мезокеуекті алюмосиликат, синтетикалық цеолит және табиғи цеолит катализаторлары қатысында жоғары температура крекинг процестері зерттелді. Катализаторлар 92,2 % дейін сұйық өнімдер түзілуіне әкеледі, шайырлы-асфальтенді қосылыстардың мөлшерін 52,4 %-дан 16,3 % дейін төмендетеді. *Қорытынды.* Беке және Мунайлы Мола кен орындарының табиғи битумдары ауыр мұнайға жатады және негізінен н-алкандарынан, терпандар мен стерандардан тұрады. Мезокеуекті алюмосиликатты катализаторды қолдану каталитикалық крекингтің тиімділігін арттырып, сұйық өнімдердің шығымын 89%-ға дейін жеткізді және жеңіл фракциялар үлесін көбейтті. Катализатордың жоғары белсенділігі ірі кеуек өлшемімен түсіндіріледі. Крекинг нәтижесінде асфальтендердегі алифатты және полинафтенді құрылымдар бұзылып, ароматтылық артады, ал оттекқұрамды қосылыстар саны азаяды; шайырларда ароматтылық нафтен сақиналары мен алкил топтарының ыдырауы есебінен өседі.

Түйінді сөздер: табиғи битум, катализаторлар, крекинг, шайырлы-асфальтенді қосылыстар, реакциялар бағыты

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КАТАЛИТИЧЕСКАЯ ПЕРЕРАБОТКА ПРИРОДНЫХ БИТУМОВ НЕФТЕБИТУМИНОЗНЫХ ПОРОД

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Резюме. *Введение.* Битумы в залежах битуминозных песчаников потенциально представляют собой очень богатый источник энергии. Вместе с тем, доступ к основной части этих запасов затруднен, и для превращения битумов в жидкие малосернистые продукты потребуются нестандартные решения, так как свойства обычной нефти и битумов существенно различаются. *Методы.* Для получения целевых продуктов из нефтебитуминозной породы сначала необходимо осуществить извлечение битумов из породы, и только после этого можно дальше перерабатывать. В настоящее время промышленная добыча битумов в Канаде ведется с применением горнотехнических методов. В связи с этим Казахстану необходимо развивать отечественные технологии, адаптированные к условиям Западного Казахстана и внутреннему спросу. *Результаты и обсуждение.* Были исследованы процессы высокотемпературного крекинга природных битумов Западного Казахстана в присутствии мезопористого алюмосиликатного, синтетического цеолитного и природного цеолитного катализаторов. Применение катализаторов обеспечило выход жидких продуктов до 92,2 % и снижение содержания смолисто-асфальтеновых соединений с 52,4 % до 16,3 %. *Заключение.* Групповой и детальный физико-химический анализ показал, что природные битумы месторождений Беке и Мунайлы Мола относятся к тяжелым нефтям и содержат н-алканы, а также ароматические углеводороды (терпаны и стераны). Для их каталитического крекинга предложен мезопористый алюмосиликатный катализатор, обеспечивающий выход жидких продуктов до 89% и повышение доли нефтяной фазы до 77% за счет увеличения легких фракций. Высокая активность катализатора обусловлена крупным размером пор. В результате крекинга разрушаются алифатические и полинафтеновые структуры асфальтенов, возрастает ароматичность и снижается содержание кислородсодержащих соединений; в смолах увеличивается ароматичность за счёт разрушения нафтеновых колец и алкильных заместителей.

Ключевые слова: природный битум, катализатор, крекинг, смолисто-асфальтеновые компоненты, направление реакции.

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