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Влияние условий гидротермального сжижения биомассы на фракционный состав и физические свойства получаемой бионефти

*Исследовано влияние двух типов катализаторов и соотношения катализатор/сырье на фракционный состав и основные физические свойства бионефти. В качестве сырья была использована биомасса микроводорослей *Chlorella*. В качестве катализатора были применены: цеолит ZSM-5 в H-форме и ZrO_2/Al_2O_3 в сульфатированной форме. Показано, что увеличение загрузки катализатора положительно влияет на содержание бензиновой и дизельной фракции в составе бионефти, а также заметно снижает ее плотность, вязкость и в незначительной степени способствует снижению содержания общей серы.*

Ключевые слова: бионефть, гидротермальное сжижение, биомасса микроводорослей, гетерогенный катализ.

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The Effect of Hydrothermal Liquefaction Conditions on Fractional Composition and Physical Properties of the Resulting Bio Crude Oil

*The effect of two types of catalysts and the ratio "catalyst / feedstock" on the fractional composition and basic physical properties of bio crude oil was investigated. *Chlorella* microalgae biomass was used as raw material. Zeolite ZSM-5 in the H-form and ZrO_2 / Al_2O_3 in the sulfated form were used as catalyst. It has been shown that an increase in catalyst loading has a positive effect on the content of gasoline and diesel fractions in bio crude oil, significantly reduces its density, viscosity, and slightly reduces the total sulfur content.*

Key words: bio crude oil, hydrothermal liquefaction, microalgae biomass, heterogeneous catalysis.

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Влияние режимных параметров пилотной установки высокотемпературного трехфазного синтеза Фишера — Тропша на основные показатели процесса

*Продемонстрирована принципиальная возможность проведения высокотемпературного трехфазного синтеза Фишера — Тропша, который осуществляется в присутствии железосодержащих каталитических дисперсий в барботажном реакторе колонного типа. Активация каталитической системы при этом проводится *in situ* в токе оксида углерода. Установлено, что размер частиц железосодержащей дисперсии, используемой в качестве катализатора указанного процесса, зависит от способа введения раствора*

прекурсора, однако во всех случаях не превышает 50 нм. Установлено, что описанный метод проведения трехфазного высокотемпературного синтеза Фишера — Тропша дает возможность достигнуть высоких показателей процесса: выход по целевым продуктам до 138 г/м³ при селективности их образования 90%. Свойства разработанного катализатора дают возможность работать длительное время при высоких конверсиях без снижения селективности процесса.

Ключевые слова: синтез Фишера — Тропша, суспензия, каталитические дисперсии, пилотная установка

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Influence of Pilot Plant Behavior Mode to the Main Process Indicators of High-Temperature Three-Phase Fischer — Tropsh Synthesis

The principal possibility of high-temperature three-phase Fischer-Tropsch synthesis, which is carried out in the presence of iron-containing catalytic dispersions in a column type bubbler reactor, is demonstrated. The activation of the catalytic system is carried out in situ in a CO stream. It was found that the particle size of the iron-containing dispersion used as a catalyst for this process depends on the method of introducing the precursor solution, but in all cases it does not exceed 50 nm. It is established that the described method for conducting three-phase high-temperature Fischer-Tropsch synthesis makes it possible to achieve high process performance: yield for target products up to 138 g/m³ with a selectivity of its formation of 90%. The properties of the developed catalyst make it possible to work for a long time at high conversions without reducing the selectivity of the process.

Key words: Fischer — Tropsch synthesis, suspension, catalytic dispersion, pilot plant.

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Особенности эжектирования газа в барботажной колонне для синтеза

Фишера — Тропша с наноразмерными суспендированными катализаторами

Впервые изучены особенности функционирования газожидкостного эжектора, могущие проявляться в трехфазном суспензионном синтезе Фишера — Тропша, использующем катализаторы с наноразмерными частицами. Для модельной барботажной установки, конструкционно воспроизводящей пилотный реактор вышеуказанного процесса, показана возможность одновременного выполнения эжектором как функций газового диспергатора колонны (при наличии дополнительных газораспределительных устройств или же без таковых), так и устройства, обеспечивающего принудительную циркуляцию жидкой среды в контуре установки в целом. Достигнут режим сверхзвукового эжектирования газа и оценено его влияние на характер барботажа.

Ключевые слова: эжектор, барботажный реактор, циркуляция суспензии, диспергирование газа.

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Peculiarities of Gas Ejection in the Bubble Column

for the Fischer – Tropsch Synthesis with Nanosized Slurry Catalysts

A possible role of the gas-liquid ejector as a gas distributor for the slurry Fischer-Tropsch bubble column reactor using nanoscale catalyst particles was first studied. Using the full-size model of the pilot-scale reactor, the possibility of simultaneous gas bubble flow generation together with forced liquid medium circulation through the bubble column and the recirculation line of the reactor was demonstrated. The supersonic gas ejection mode was reached and its effect on the explicit bubbling mode was discussed.

Key words: *ejector, bubble column reactor, slurry circulation, gas distribution.*

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Исследование галлуазита в качестве компонента цеолитных катализаторов конверсии диметилового эфира в углеводороды

Исследованы текстурные и кислотные свойства цеолитных катализаторов HZSM-5 с применением в качестве связующего компонента Al₂O₃ и алюмосиликатных нанотрубок галлуазита. Изучена стабильность и каталитическая активность образцов в реакции конверсии диметилового эфира в углеводороды. Показано, что в сравнении Al₂O₃ применение галлуазита приводит к перераспределению продуктов в сторону образования углеводородов C₅–C₈.

Ключевые слова: галлуазит, диметиловый эфир, синтез углеводородов, HZSM-5.

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Study of Halloysite as a Component of Zeolite Catalysts

for Dimethyl Ether to Hydrocarbons Conversion

The textural and acid properties of HZSM-5 zeolite catalysts were studied using Al₂O₃ and halloysite as a binders. The stability and catalytic activity of the samples in the conversion of dimethyl ether to lower olefins was studied. It was shown that, in comparison with Al₂O₃, the use of halloysite leads to a redistribution of products towards the formation of C₅–C₈ hydrocarbons.

Key words: *halloysite, dimethyl ether, hydrocarbon synthesis, HZSM-5.*

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Влияние природы загустителя на свойства полимочевинных смазочных композиций на основе сложных эфиров

Синтезированы полимочевинные смазочные композиции, имеющие условно две и четыре мочевиновые группы на молекулу загустителя полимочевины, в присутствии второго биоразлагаемого загустителя (наноцеллюлозы, цетарилового спирта). Оценены такие свойства, как температура каплепадения, пенетрация, коллоидная стабильность, реологические, трибологические и противоизносные свойства. Установлено, что свойства смазочных композиций меняются в зависимости от количества мочевиновых групп на молекулу полимочевины и содержания второго биоразлагаемого загустителя. Смазочные композиции на основе тетрамоуевины обладают меньшей вязкостью, лучшими трибологическими и противоизносными свойствами независимо от содержания наноцеллюлозы, что подтверждает решающее влияние структуры мочевинового загустителя на свойства смазочных композиций.

Ключевые слова: полимочевина, смазочные композиции, наноцеллюлоза, олеогели, сложноэфирная основа, трибологические свойства, реологические свойства.

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Effect of Thickener Nature on the Properties of Polyurea Ester-Based Greases

In this study, polyurea lubricant compositions were synthesized. They had conditionally two and four urea groups per molecule of a polyurea thickener and were synthesized including in the presence of a second biodegradable thickener (nanocellulose or cetaryl alcohol). Such properties as dropping point, penetration grade and oil separation as well as rheological, tribological and antiwear properties were evaluated. It is established that the properties of lubricating compositions vary depending on the number of urea groups per polyurea molecule and the content of the second biodegradable thickener. Tetraurea-based greases exhibit lower viscosity, better tribological and anti-wear properties regardless of the nanocellulose content, which shows the decisive influence of the polyurea structure on the properties of the greases.

Key words: polyureas, lubricating greases, nanocellulose, oleogels, ester oil, tribology, rheology.

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Улучшение триботехнических свойств полимочевинных смазок на разных дисперсионных средах с применением различных наполнителей

В работе рассмотрены свойства наполнителей для пластичных смазок, таких как, дисульфид молибдена, дисульфид вольфрама и графит. Изучено их влияние на триботехнические свойства полимочевинных пластичных смазок, приготовленных на различных дисперсионных средах, с помощью трибометра Бруггера, в соответствии со стандартом DIN 51347-1:2000-0 и трибометра SRV-3 в соответствии со стандартом ASTM D5707.

Ключевые слова: пластичные смазки, полимочевинные смазки, добавки к смазкам, наполнители, дисульфид молибдена, графит, дисульфид вольфрама, трибологические свойства смазок.

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Improving of Tribotechnical Properties of Polyurea Greases

Based on Different Dispersion Media with Different Fillers

This work describes the properties of the fillers in lubricating greases, such as molybdenum disulfide, tungsten disulfide and graphite. Their influence on the tribotechnical properties of polyurea grease, enhanced with various dispersion media, was studied using the Brugger tribometer in accordance with DIN 513471: 20000, SRV-3 tribometer in accordance with ASTM D5707.

Key words: greases, polyuria grease, solid additives, fillers, molybdenum disulfide, graphite, tungsten disulfide, tribological properties of greases.

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Получение низкосернистого судового топлива

Представлены результаты испытания системы катализаторов, разработанной для деме­таллизации и обессеривания смеси вакуумного газойля и тяжелого газойля коксования при давлении 6 МПа, температуре 360–370°C и объемной скорости подачи сырья 1 ч⁻¹ с получением продукта, содержащего 0,05% мас. серы. Разработан состав судового топлива марки RMB 30 с вовлечением дистиллятных фракций вторичных процессов и депрессорно-диспергирующей присадки для доведения температуры текучести до требований ГОСТ 32510–2013.

Ключевые слова: вакуумный газойль, тяжелый газойль коксования, деме­таллизация, гидрообессеривание, депрессорно-диспергирующая присадка, низкосернистое судовое топливо.

L. A. Gulyaeva, M. M., Lobashova, T. N. Mitusova, O. I. Shmel'kova, V. A. Khavkin, P. A. Nikulshin,

All-Russian Research Institute of Oil Refining JSC

Production of Low-Sulfur Marine Fuel

Results of testing of catalyst developed for demetallization and desulfurization of vacuum gasoil and heavy coking gasoil mixture at 6 MPa hydrogen pressure, 360–370°C temperature, 1 h⁻¹ liquid hourly space velocity (LHSV) to obtain a product containing sulfur less 0.05% wt. are presented. The composition of RMB 30 marine fuel with the involvement of distillate fractions of secondary processes and depressor-dispersing additive to bring the flow temperature to the requirements of GOST 32510-2013 was developed.

Key words: vacuum gasoil, heavy coking gasoil, demetallization, desulfurization, depressor-dispersing additive, low-sulfur marine fuel.

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Влияние микроволновой обработки товарной тяжелой нефти на ее текучесть

В работе экспериментально исследовано воздействие нагрева товарной тяжелой нефти сверхвысокочастотным (СВЧ) электромагнитным полем на изменения ее вязкости. Исследовано влияние фазового состояния надмолекулярных образований на реологические свойства тяжелой нефти после диэлектрического нагрева. Выполнена оценка энергии межмолекулярного взаимодействия асфальтеновых наноагрегатов различных масштабных уровней на базе реометрических исследований тяжелой нефти, обработанной СВЧ-полем. Установлено, что для стабильной по асфальтенам нефти наиболее эффективной является гибридная схема микроволновой обработки.

Ключевые слова: тяжелая нефть, ассоциативная наножидкость, СВЧ-нагрев, гибридная схема нагрева, эффективная вязкость, фазовое состояние, энергия активации вязкого течения.

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Influence of Microwave Processing of Heavy Stock Oil on Oil Fluidity

The paper experimentally investigated the effect of heavy stock oil heating by ultra-high frequency (microwave) electromagnetic field on oil viscosity changes. There has been studied an influence of the supramolecular formations phase state on the rheological properties of heavy oil after various dielectric heating schemes application. The energy of the intermolecular interaction of asphaltene nanoaggregates of various scale levels is estimated on the basis of rheometric studies of heavy oil treated with microwave energy. It has been established that for asphaltene stable oil the hybrid microwave processing scheme is the most effective.

Key words: heavy oil, associative nanofluid, microwave heating, hybrid heating scheme, effective viscosity, phase state, viscous flow activation energy.

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Производные фурфурола в качестве компонентов топлив

В статье показана возможность применения производных фурфурола, полученных каталитическим гидрированием на медных и никелевых катализаторах с преобразованием фурфурола до топливных компонентов. Было оценено их влияние на антидетонационные свойства и химическую стабильность. Отмечено, что фураны проявляют свою наибольшую эффективность в низкооктановых углеводородных базах, как например, бензин гидрокрекинга при содержании 5–30% мас. Высокая концентрация потенциальных смол может привести к образованию отложений в камере сгорания двигателя, что, в свою очередь, требует проведения дополнительных исследований.

Ключевые слова: фурфурол, 2-метилфуран, фурфуриламмин, дипропилацеталь фурфурола.

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Furfural Derivatives as Fuel Components

Furfural derivatives, prepared from vegetable raw materials, have recently become popular as high-performance fuel additives. Special attention is drawn to these compounds as oxygenates. A lot of research have been developed for preparing new materials from furfural, but it still doesn't clear about their effect on physicochemical properties of fuels. In this article is shown the possibility of adding furfural derivatives, which are obtained by their hydrogenation at copper and nickel catalysts with full furfural conversion to fuel. Their impact on antiknock properties and chemical stability was evaluated. It was noticed that furans are mostly effective in low-octane bases (gasoline HC) at 5–30 % wt. concentration. High concentration of potential gum may lead to carbon depositions in the combustion chamber of an engine that requires additional tests.

Key words: furfural, 2-methylfuran, furfurylamine, furfural dipropyl acetal.

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Композиции ацеталей фурфурола как комплексная добавка к дизельным топливам

Рекомендован метод получения дибутил- и диамилацеталей фурфурола на лабораторной установке проточного типа. Определены цетановые числа композиций с бутанолом и при введении дикумилпероксида, низкотемпературные свойства и седиментационная устойчивость дизельных топлив при введении 3% мас. образцов ацеталей. Оценены смазывающие свойства исследуемых образцов ацеталей и определены численные значения диаметра пятна износа.

Ключевые слова: фурфурол, ацетали, цетановое число, сульфокатионит, смазывающая способность.

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Furfural Acetal Compositions as an Complex Additive to Diesel Fuels

Preparation method for furfural dibutyl and diamyl acetals in flow-type laboratory setup is recommended. Cetane numbers of compositions with butanol and dicumyl peroxide addition, low-temperature properties and sedimentation stability of diesel fuels with 3% wt. acetals were determined. Lubricating properties of the studied acetals were evaluated and values of wear spot diameter were determined.

Key words: *furfural, acetals, cetane number, sulphocationite, lubricity.*

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Фазово-переходные материалы в энергетике: современное состояние исследований и перспективы применения

В работе представлен обзор современных исследований в области фазово-переходных материалов, перспективных для снижения энергопотерь в промышленных условиях и в системах отопления/кондиционирования жилых помещений. В частности, рассмотрены проблемы внедрения данных технологий, такие, как стратегия инкапсулирования активного вещества, стабильность полученных материалов с фазовым переходом и возникающие коррозионные осложнения. Кроме того, фазово-переходные материалы могут использоваться в холодильных системах для повышения КПД, а также в хранилищах для снижения технологических потерь, связанных с испарением топлив и технических жидких продуктов. Выделены перспективные направления использования данных материалов и намечены пути решения возможных проблем.

Ключевые слова: фазово-переходные материалы, хранение тепловой энергии, плавление, кристаллизация, переохлаждение, инкапсуляция.

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Phase Change Materials in the Energy Sector: Current State of Research And Application Prospects

In this work, we review the recent studies on phase change materials promising to reduce energy losses in industrial conditions and domestic heating/air conditioning systems. Particularly, we focus on the challenges of phase change materials application, such as a strategy for the encapsulation of active substance, the stability of obtained materials with a phase transition, and emerging corrosion complications. Phase change materials could be employed in refrigerating systems to increase thermal efficiency, and in the storage facilities to reduce evaporative losses of fuels and industrial liquid products as well. Promising areas of using these materials are highlighted and ways to solve possible problems are outlined.

Key words: *phase change materials, thermal energy storage, melting, crystallization, subcooling, encapsulation.*

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A New Reservoir Protection Technology for Offshore Condensate Gas Wells During Shut-in

Wells in tight sandstone condensate gas reservoir of East China Sea often suffer from dramatic production decline after being closed for several days. The closure can be caused by typhoon. For example, the gas production of target well B₁ studied in this paper plummeted from 50,000 to 0 m³/d after the typhoon. This study aims to propose a reservoir protection technology preventing the offshore condensate gas wells from production decline caused by emergent shut-in operations. Firstly, based on comprehensive analysis of the production parameters, experimental data, and process mechanism it was shown that the sudden drop in gas production of well B₁ is attributed to wellbore liquid loading and reservoir liquid locking damage and is not caused by the insufficient recoverable reserves and solid plugging damage. Secondly, a liquid locking removal technology combining wettability alteration treatment with coiled tubing gas lift technique was proposed and applied on the well B₁. The result showed that the gas production increased dramatically from 0 to 40,000 m³/d. However, even though the liquid locking removal technology proved to be efficient, it is also time-consuming and costly. Therefore, in order to optimize the construction time and costs for the conditions of the actual offshore production, the improved innovative two-step reservoir protection technology was suggested, combining foam drainage gas recovery and wettability alteration treatment.

Keywords: Offshore condensate gas reservoir, typhoon, emergent shut-in operation, reservoir protection technology.

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Analysis of Seepage Characteristics of Complex Pore Structure Rock by Digital Core Method

Application of traditional rock physics experimental methods in investigating the macroscopic petrophysical properties of low porosity, low permeability, and high complexity pore structure reservoirs is limited by the high cost,

long cycle, large error, and the specific difficulties of such reservoirs. It is also difficult to evaluate quantitatively the influence of microscopic reservoir structure on rock physical properties. Application of micro-CT scanning and advanced imaging processing technology enables to establish an accurate representative pore space model equivalent to the true core structure. The numerical simulation with Avizo-XLab solver method was used to calculate the pore fluid flow based on the Navier-Stokes equation and the Darcy seepage law. The visualization method helped to display the pore-space fluid flow in a three-dimensional streamline, and the pore pressure distribution field is displayed, making the simulation results clear and intuitive. The research shows that numerical simulation based on the digital platform provides instrument for the measurement of the fluid micro flow and the rock seepage parameters. This paper provides a new method for digital research on rock physical properties which helps to overcome the deficiency of traditional rock physics experimental research.

Keywords Traditional rock physics experiments, microscopic factors, micro-CT scanning, micro flow of pore fluid, rock seepage parameters.

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Kinetics Study of Surface Reaction between Acid and Sandstone based on the Rotation Disk Instrument

The chemical reaction between acid and rock mineral during the acidizing process is the key point for the acidizing technique. In this study a rotating disk method is used to simulate strata conditions, such as high temperature, high pressure, and acid shear flow reaction. The work investigates the kinetics of the rock acidizing reaction and analyses the factors affecting the reaction rate, including mineral composition, temperature, pressure, acid concentration, shear velocity, and common-ion effect. The kinetic parameters, such as velocity constant, reaction order, activation energy, and frequency factor, were successfully extracted by curve fitting and linear regression method. It was shown that hydrochloric acid does not participate directly in the reaction between mud acid (which is the mixture of hydrochloric and hydrofluoric acids) and sandstone mineral, but catalyses the reaction between hydrofluoric acid and sandstone. In this work, the acid rock reaction rate equation has been established, and the impact of the hydrochloric acid catalytic effect has been considered.

Keywords: acidizing, acid-rock reaction rate, sandstone, reaction kinetics, rotating disk apparatus.

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Method of Fractal Reservoir with Irregular Crack Bedrock System and its Application for Well Test Analysis

The chemical reaction between acid and rock mineral during the acidizing process is the key point for the acidizing technique. In this study a rotating disk method is used to simulate strata conditions, such as high temperature, high pressure, and acid shear flow reaction. The work investigates the kinetics of the rock acidizing reaction and analyses the factors affecting the reaction rate, including mineral composition, temperature, pressure, acid concentration, shear velocity, and common-ion effect. The kinetic parameters, such as velocity constant, reaction order, activation energy, and frequency factor, were successfully extracted by curve fitting and linear regression method. It was shown that hydrochloric acid does not participate directly in the reaction between mud acid (which is the mixture of hydrochloric and hydrofluoric acids) and sandstone mineral, but catalyses the reaction between hydrofluoric acid and sandstone. In this work, the acid rock reaction rate equation has been established, and the impact of the hydrochloric acid catalytic effect has been considered.

Keywords: acidizing, acid-rock reaction rate, sandstone, reaction kinetics, rotating disk apparatus.

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Methodology for Estimating Strength and Elasticity of Granular Profile Control Agents used in ASP Water Flooding

A new method for estimating the strength of granular profile control agents during ASP water flooding was proposed considering the transport behavior of granular profile control agents in the reservoir. The strength of granular profile control agents (size over 80 mesh) passing through the sieve (diameter 0.1 mm) is determined by the value of the breakthrough pressure. The strength of profile control agent particles (size 20-60 mesh) is determined by the breakthrough pressure value, measured on a sieve with a diameter of 0.3 mm. Breakthrough pressure values of 8 types of granular profile control agents were determined under various experimental conditions (for particle expansion time of 24 hours, 120 hours, and 30 days, respectively). The results show that the longer the expansion time of granular profile control agents, the smaller the strength. According to the results of a study conducted on a granular profile control agent of the same type (for example: QC5050) with different particle sizes (80-120 mesh, 80 mesh and 20-40 mesh), it can be concluded that the larger the size of the particles of the profile control agents, the greater the value of the corresponding breakout pressure. A new parameter has been introduced - a rebound coefficient - for evaluating elasticity of granular profile control agents. The rebound coefficient was measured for 8 types of granular profile control agents at different pressures (2, 4, 6 and 8 MPa). The results show that the number of samples with a rebound ratio higher than 20% reached more than 70% in 64 experiments conducted on 8 samples. A negative correlation relationship between the rebound coefficient and the breakthrough pressure was determined, and a method was

proposed for estimating the strength and elasticity of the granular profile control agents considering the influence of the rebound coefficient and the breaking pressure through the sieve.

Keywords: granular profile control agents, estimation of the strength and elasticity, rebound coefficient, breakthrough pressure

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Pore Pressure and Stress Distribution around the Wellbore in Shale Formation Based on the Generalized Plane Strain Principles

The article presents a new finite-element model for investigating the pore pressure and stress distribution around the wellbore in shale formation. The model which is based on generalized plane strain principle is suitable for any directional well in the anisotropic formation relative to analytical and plane strain model. Meanwhile, the model is more effective than three-dimensional model. The FEM model takes thermal and chemical effects into consideration, so a nonlinear thermo-chemo-poroelastic coupling theory is used to analyze the problem. It is proved that the present model is validated well with the existing analytical model. The effects of the thermal, chemical potential and elastic anisotropy on pore pressure and stress distribution are discussed. It is illustrated that lower temperature and chemical potential could prevent pore pressure and effective radial raising, which are helpful to reduce the risk of wellbore failure. The influence of Poisson's ratio anisotropy to pore pressure and effective stress is far slighter than Young's modulus.

Keywords: generalized plane strain; pore pressure; effective stress; thermo-chemo-poroelastic; wellbore stability.

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Simulation of Mechanism of Hydraulic Fracture Propagation in Fracture-Cavity Reservoirs

The acid fracturing is a key measure to increase production of fracture-cavity reservoirs. Affected by fracture-cavity system hydraulic fractures will not propagate in a plane and the mechanism of hydraulic fracture propagation is complicated. Therefore, considering the characteristics of hydraulic fracture propagation in fracture-cavity reservoirs, we established an extended finite element (XFEM) model for hydraulic fractures in fracture-cavity reservoirs. The simulation discussed hydraulic fracture extension in cases of a single cave and a single natural fracture, and revealed the mechanism of dynamic propagation and extension of hydraulic fractures. The results indicated severe stress concentrations near caves, resulting in deflections of the fracture propagation direction. In case of a single cave, upon shifts of shafts from the central line of the cave, the conditions of penetration of the cave by hydraulic fractures were investigated. It was shown that in case of small approaching angles, the hydraulic fractures tend to deflect and join

natural fractures; also, the hydraulic fractures can deflect and join natural fractures with large approaching angles and weak cementation. Generally, small approaching angles and long natural fractures tend to induce hydraulic fractures and assist propagation towards the caves, thus increasing the probability of cave penetration.

Keywords: Hydraulic fracture, carbonate reservoir, fracture propagation, extended finite element, approaching angle.