Recycling of waste plastics to liquid fuel mixture over composite zeolites catalysts

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Plastic waste production and consumption is increasing at an alarming rate with the increase of the human population, rapid economic growth, continuous urbanization, and changes in lifestyle. In addition, the short life span of plastic accelerates the production of plastic waste on a daily basis. Plastic waste recycling is carried out in different ways, but in most developing countries, open or landfill disposal is a common practice for plastic waste management. Plastic recycling into feedstocks, also known as chemical recycling, is encouraged all over the world. One such area is the thermal and catalytic thermal degradation of plastics into hydrocarbon fractions, which can be used as high-quality motor fuel after appropriate processing. Hydrocracking in the presence of a catalyst is a promising method of converting waste plastic materials to high quality liquid transportation fuels with decreased amounts of olefins and heteroatoms such as S, N, Cl, N, and O.

The article deals with the study of hydrocracking of waste plastic into high quality liquid fuel on various catalysts based on natural zeolite deposits Taizhuzgen. The aim of the work is to determine the effect of new composite catalysts on the yield of liquid products by studying the specific surface and porous structure based on natural zeolite modified with Mo salt. It is established that the modification of natural zeolite with Mo affects the morphology of the catalyst, therefore, the obtained catalysts have different effects on the yield and composition of liquid fractions during the hydrogenation thermocatalytic transformation of hydrocarbons. The highest yield of liquid products (61.56%) was achieved using the 2% Mo/Taizhuzgen zeolite catalyst, which was chosen as optimal.

Keywords: polymer wastes; hydrogenation thermocatalytic recycling; zeolite; morphology of catalysts; hydrocracking.

Пластмасса қалдықтарын сұйық отынға композитті цеолитті катализаторлар қатысында өңдеу

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Пластикалық қалдықтарды өндіру және тұтыну халық санының ұлғаюымен, экономиканың қарқынды өсуімен, үздіксіз урбанизациялаумен және өмір салтындағы өзгерістермен артуда. Сонымен қатар, пластик заттарды қолдану пластикалық қалдықтардың артуына әкеледі. Пластикалық қалдықтарды қайта өңдеу әр түрлі тәсілдермен жүзеге асырылады, бірақ дамушы елдердің көпшілігінде ашық немесе үйінділі кәдеге жарату пластикалық қалдықтармен жұмыс істеудің әдеттегі тәжірибесі болып табылады. Пластикті шикізатқа қайта өңдеу, сонымен қатар одан пайдалы мотор отындарын алу негізгі мәселелердің бірі болып табылады. Мұндай салалардың бірі – пластмассаның көмірсутегі фракцияларына термиялық және каталитикалық термодеструкциясы және өңдеуден кейін жоғары сапалы мотор отыны ретінде пайдаланылуы. Гидрокрекинг процесі Тайжүзген табиғи цеолиті негізіндегі катализатордың қатысуымен пластмасса қалдықтарын S, N, Cl, N және O сияқты төмен олефиндер мен гетеротомдары бар жоғары сапалы сұйық көлік отындарына айналдырудың перспективалық әдісі болып табылады.

Мақала Тайжүзген табиғи цеолит кен орындары негізіндегі түрлі катализаторлар қатысында пластмассалардың гидрокрекингін зерттеуге және жоғары сапалы сұйық отын алуға негізделген. Жұмыстың мақсаты Мо тұзымен модификацияланған табиғи цеолит негізіндегі жаңа композитті катализаторлардың меншікті беті мен кеуекті құрылымын зерттеу арқылы сұйық өнімдердің шығымына әсерін анықтау болып табылады. Табиғи цеолитті Мо тұзымен модификациялау катализатордың морфологиясына әсер ететіні анықталды, сондықтан алынған катализаторлар көмірсутектердің гидрогенизациялық термокаталитикалық өңдеуде сұйық фракциялардың шығымы мен құрамына әр түрлі әсер етеді.

Түйін сөздер: полимер қалдықтары; гидрогенизациялық термокатализдік өңдеу; цеолит; катализатор морфологиясы; гидрокрекинг.

Переработка отходов пластмасс в жидкое топливо на цеолитных катализаторах

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Производство и потребление пластиковых отходов растет тревожными темпами с увеличением численности населения, быстрым экономическим ростом, непрерывной урбанизацией и изменениями в образе жизни. Переработка пластика в сырье, также известное как химическая переработка, поощряется во всем мире. Одной из таких областей является Термическая и каталитическая термодеструкция пластмасс на углеводородные фракции могут быть использованы в качестве высококачественного моторного топлива после соответствующей переработки. Гидрокрекинг в присутствии катализатора является перспективным методом превращения отходов пластмасс в высококачественные жидкие транспортные топлива с пониженным содержанием олефинов и гетероатомов, таких как S, N, Cl, N и O.

Статья посвящена исследованию гидрокрекинга отработанных пластмасс в высококачественное жидкое топливо на различных катализаторах на основе природных цеолитов месторождений Тайжузген. Целью работы является определение влияния новых композитных катализаторов на выход жидких продуктов путем исследования удельной поверхности и пористой структуры на основе природного цеолита, модифицированного солью Мо. Установлено, что модификация природного цеолита солью Мо оказывает влияние на морфологию катализатора, поэтому полученные катализаторы по-разному влияют на выход и состав жидких фракций при гидрогенизационном термокаталитическом превращении углеводородов.

Ключевые слова: полимерные отходы; гидрогенизационная термокаталитическая переработка; цеолит; морфология катализаторов; гидрокрекинг.
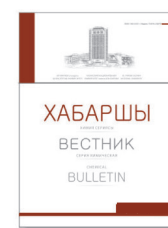
Recycling of waste plastics to liquid fuel mixture over composite zeolites catalysts

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1. Introduction

Plastics play an important role in our daily life, and our ever-growing dependence on them is justified by their versatility, low weight and low cost of production compared to other classic materials such as wood, concrete and metal [1]. The production of plastic materials such as polyethylene (PE), polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET) and polyvinyl chloride (PVC) is increasing every year [2] due to their consumption in packaging, construction, agriculture, electrical and electronic devices and healthcare applications [3].

The current state of raw material processing, also known as chemical processing or tertiary processing, is aimed at converting polymer waste into original monomers or other valuable chemicals. These products are useful as raw materials for a variety of downstream industrial processes or as transportation fuel. There are three main approaches: depolymerization, partial oxidation, and cracking (thermal, catalytic, and hydrocracking) [4]. Thermal degradation of plastics is of great concern in connection with the processing of raw materials, as they are converted into gas, liquid (oil) and solid products, each of which can be used as a source of energy and/or chemical raw materials. Plastic waste can be a valuable alternative source of energy production [5,6]. The composition and yield of pyrolysis products directly depend on the reaction conditions (temperature, heating rate, pressure, presence of a catalyst, etc.), as well as on the structure of the aspirated material [7-10]. The interaction between materials in food waste has a significant impact on the selection of individual components of the liquid product [11-13].

Catalysts play an important role in hydrocracking of plastics. A bifunctional catalyst is usually needed for this purpose. The presence of an acidic function, as well as a hydrogenation-dehydrogenation function, is usually necessary to achieve significant conversion, high yield and high quality of liquid products, and to reduce the amount of coke deposits [3]. It is well known that porous materials such as zeolites, carbon, meso-porous materials, and other molecular sieve materials are one of the most popular catalytic materials that are widely used in the oil refining and chemical industries. Due to the rapid reduction of workings and the high demand for environmental protection, it is urgent to develop new high-performance catalysts, especially with porous materials [14]. At a lower temperature (400°C) virtually similar results were obtained with both catalysts, while at a higher temperature, 430°C, the Mo-containing zeolite catalyst was more active in converting polyethylene to reaction products [15,16]. New catalysts based on the natural zeolite of the Taizhuzgen Deposit modified by Mo (VI) salt will be actively and selectively used for the process under study [17].

The aim of the work is to study the effect of new composite catalysts on the yield of liquid products by studying the specific surface area and porous structure based on natural zeolite modified modified by Mo (VI) salt [18].

2. Experiment

2.1 Preparation of catalysts

Zeolite at the "Taizhuzgen" field was crushed with a sieve up to 0.25 mm in size. Zeolite (10 g) was mixed with 500 mL of 1M NH_4Cl solutionon a water bath using a mechanical stirrer for 18 h (three times for 6 h). After washing the Taizhuzgen zeolite with NH_4Cl, NH_4^+ and Cl^- ions remain inside the zeolite. Zeolite was filtered from NH_4Cl solution and washed with distilled water till the compete removal of Cl^-ions. Absence of chlorine

ions was confirmedusing a reaction with a 0.1 mol/LAgNO$_3$. Filtered zeolite was dried till the constant mass in a drying oven SNOL 75/350 (AB UMEGA, Lithuania) at 100°C.To remove ammonia gas and keep H$^+$ ions, zeolite was heated in the reactor at 100°C (1 h), 200°C (1 h), 300°C (1 h) and 500°C (1 h).

To prepare four catalysts with a different Mo concentration (0.5, 1.0, 1.5 and 2.0% w/w), different volumes of a solution of (NH$_4$)$_6$Mo$_7$O$_{24}$·4H$_2$O were added to zeolite, which was then dried at 100°C till the constant mass and stored in a desiccator with CaCl$_2$.

2.2 Hydrogenation

The hydrogenation was carried out in the installation (Figure 1) operating in a periodic mode under a pressure of 0.5 MPa, at a temperature of up to 450°C andcontinuous stirring for 15 min. The installation consisted of a stainless steel (X18N10T) reactor with a volume of 150 mL. The reactor was heated using an alternating current heater controlled by a transformer and an ammeter. The temperature control in the reactor was carried out using a chromel-drop thermocouple. The readings were recorded on the KSP-4 device with a calibrated scale for the boiling points of water (100°C), melting of tin (232°C), lead (327°C) and zinc (427°C). Thepressure in the hydrogenation unit was created using nitrogen and measured using a gauge. A mixture of the polymer (7 g), a catalyst (0.28 g) and the paste-forming agent (7 g) was loaded into the reactor, then the reactor was checked for tightness, purged with nitrogen, a pressure of 0.5-0.6 MPa was set, after which the heating device was turned on. Caps for the plastic bottles produced by Bericap Kazakhstan LLP, collected from drinks produced by the Coca-Cola Almaty Bottlers LLP) and crushed to the particle size of 3-6 mm were used as a polymer material. Heavy residue of the crude oil from Kumkol petroleum reservoir after its distillation to 350°C was used the paste-forming agent.

Using a pressure gauge during hydrogenation, a pressure change was recorded due to an increase in temperature, gas separation and the presence of volatile components. The reactor heating was turned off 30-40 min after starting the process (when the pressure decreased), and the system was cooled to room temperature. The gas formed in the process was collected in a gasometer with a saturated NaCl solution. The amount of gas formed was calculated from the pressure difference.

The distillation of the liquid product with separation to different temperature (0-180°C, 180-250°C, 250-320°C) fractions was carried out at the installation (Figure 1). For this purpose, a condenser with a receiver was connected to the nozzle of the reactor cooled to room temperature. During the distillation, the reactor was heated, and different temperature fractions were collected. After cooling the unit, the remaining residue was removed from the reactor.

Each experiment was conducted three times under the same conditions. The deviation between the results of the experiments did not exceed 2%.

2.3 Characterization

Samples of activated zeolite and Mo(VI)/zeolite catalysts were analyzed on the JSM-6460LV (JEOL, Japan) scanning electron microscope.

Analysis of liquid distillates using gas chromatography with mass spectrometric detection (GC-MS) was performed on the 6890N/5973N (Agilent, USA) instrument. Liquid sample (0.2 µL) was introduced at 500:1 split and 240°C to the HP-INNOWax (Agilent, USA) 30 m x 0.25 mm column (film thickness 0.25 µm). Oven temperature was programmed from 50°C (maintained for 5 min) to 240°C (maintained for 10 min). Total analysis time was 34 min. Detector scanned ions in *m/z* range 10-350 amu. Identification of peaks was conducted using NIST'08 mass spectral library.
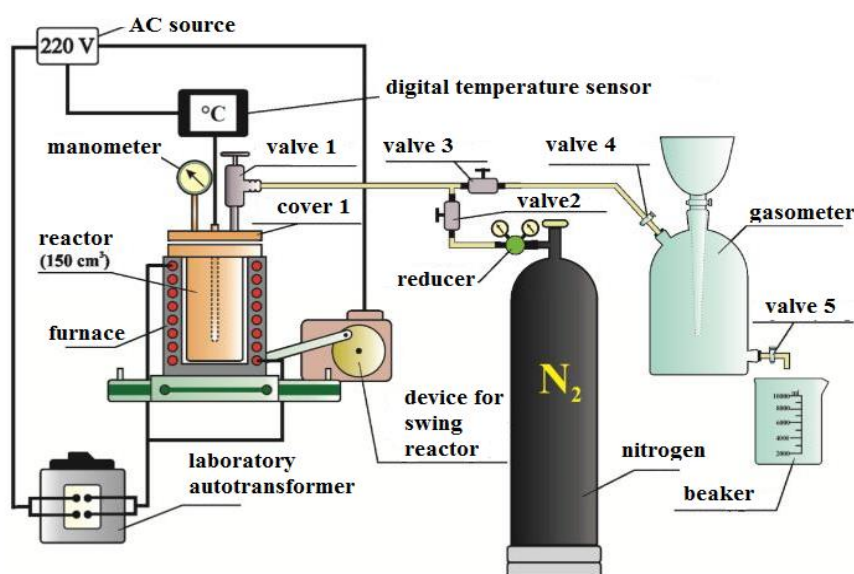

Figure 1 – Scheme of the installation used for catalytic hydrogenation of polymer waster [19]

Table 1 – Chemical composition of Taizhuzgen zeolite, %

SiO_2	TiO_2	Al_2O_3	Fe_2O_3	FeO	MnO_2	MgO	CaO	Na_2O	K_2O	SO_2	H_2O
65.15	0.23	14.25	0.90	0.54	0.10	0.74	3.16	2.17	2.74	0.05	9.97

Table 2 – Elemental composition of Taizhuzgen zeolite

| Element	O	Si	Al	K	Ca	Fe	Na	Mg	Ti	Mn
Concentration, mass.%	49.26	34.62	7.62	3.11	1.78	1.49	1.30	0.51	0.19	0.12

3. Results and Discussion

The main chemical compounds of natural zeolites are SiO_2 and Al_2O_3. Y-type zeolites have SiO_2/Al_2O_3 ratio about 2-3, X-type zeolite -3-6. The ratio of oxides of Taizhuzgen natural zeolite corresponds to the X type: SiO_2/Al_2O_3=4.57 (Table 1), and it is an active catalyst for catalytic cracking [18]. Elemental composition of Taizhuzgen zeolite contain Si, Al, K, Ca, Fe, Na, Mg, Ti, Mn whichareactive valence transition metals (Table 2).

SEM images of activated Taizhuzgen zeolite and 2% Mo/Taizhuzgen zeolite catalyst are presented in Figures 2 and 3, respectively. The sizes of most particles of activated zeolite were 711.4-1820 microns. The catalyst based on 2% Mo(VI)/zeolite had uneven surface morphology of small particles – size of most particles ranged from 755.6 to 1800 nm. In general, large-dimensional granular properties are observed for the catalyst particles. Modification of the activated zeolite surface leads to edge alignment and uneven porosity. Consequently, the zeolite particles are enlarged as a result of the modification with molybdenum (VI).

In this research, the effect of the Mo concentration in the catalyst on the yield of products and products of the process of hydrogenation of polymer waste was studied. According to the experimental results (Table 3), the highest yield of liquid products (61.56%) is achieved using the 2% Mo/Taizhuzgen zeolite catalyst. The highest yield of the lightest fraction
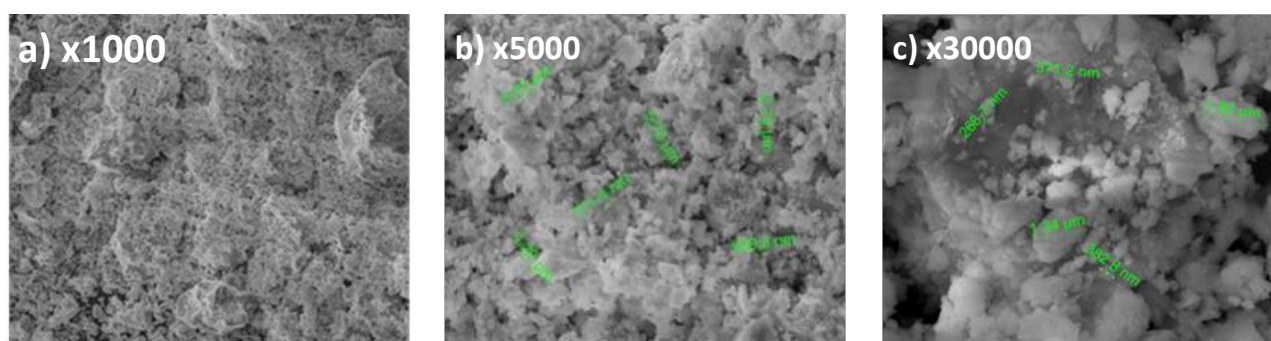
Figure 2 – SEM micrographs of activated Taizhuzgen: a) x1000; b) x5000; c) x30000
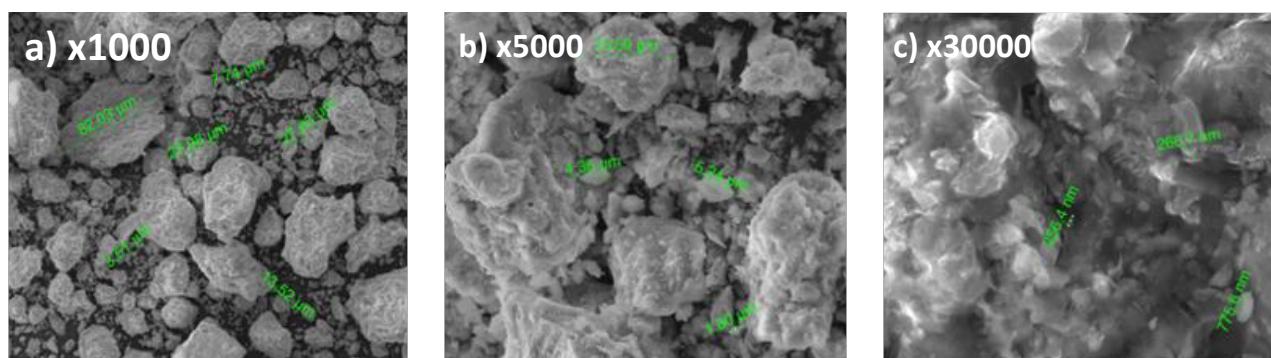
Figure 3 – SEM images of 2 % Mo/Taizhuzgen zeolite catalyst: a) x1000; b) x5000; c) x30000

(<180°C) was achieved using the catalyst containing 1% Mo. Catalyst containing 1.5% Mo provided the highest yield of the 180-250°C fraction – 21.44%. Losses during the experiments were caused by the evaporation of water from the composition of the starting materials, as well as the distillation of synthetic liquid products. Thus, 2% Mo/Taizhuzgen zeolite catalyst was used as an optimal catalyst because it provided the highest yield of liquid products.

The group hydrocarbon content of fractions with a boiling point <180°C, 180-250°C, 250-320°C obtained after thermocatalytic hydrogenation treatment of polymer wastes in the presence of 2% Mo/Taizhuzgen zeolite catalyst was compared (Figure 4). Highest concentrations of alkanes (54.9%) and aromatics (27.13%) were determined in the fraction 250-320°C, isoalkanes (33.5%) – in the fraction 180-250°C, alkenes (6.1%), cycloalkanes (22.0%) and cycloalkenes (5.35%) – in the fraction <180°C.

The material balance was calculated under optimal conditions of in the hydrogenation thermocatalytic processing of polymer residue and Kumkol fuel oil (Table 4).
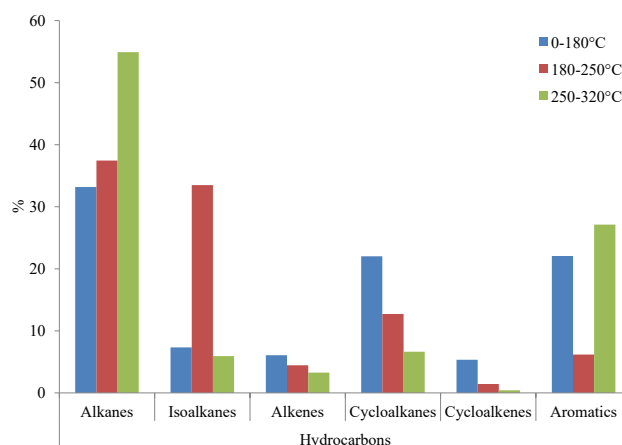

Figure 4 – Composition of distillates obtained using 2% Mo/ Taizhuzgen zeolite catalyst

Table 3 – Characteristics of products obtained in the process of thermocatalytic hydrogenation of polymer waste

Catalyst	Yield of liquid products, mass.%				P_{max}, MPa	Yield of gases, mass.%	Yield of solid waste, mass.%	Loss, mass.%
	<180°C	180-250°C	250-320°C	Σ_c				
0.5% Mo/Taizhuzgen zeolite	13.21	14.11	7.27	34.59	3.8	18.19	13.13	34.09
1% Mo/Taizhuzgen zeolite	17.52	8.79	6.58	32.89	5.9	18.19	10.91	38.01
1.5% Mo/Taizhuzgen zeolite	15.07	21.44	6.74	53.25	6.2	23.26	10.50	12.99
2% Mo/Taizhuzgen zeolite	12.44	19.12	30.00	61.56	5.1	15.31	10.80	12.33

Table 4 – Material balance of the process with 2% Mo/Taizhuzgen zeolite

No	Income:	Weight, g	Mass.%	Yield:	Weight, g	Mass.%
1	Plasticwastes	7.00	50.0	Fraction 0-180°C	1.75	12.44
2	Fuel oil	7.00	50.0	Fraction 180-250°C	2.68	19.12
				Fraction 250-320°C	4.20	30.00
				Gas	2.14	15.31
				Solid residue	1.51	10.80
				Loss	1.72	12.33
	Total	14.00	100	**Total**	14.00	100

4. Conclusions

Thus, among the studied catalysts for the process of thermocatalytic hydrogenation of polymer waste, 61.56% was a maximum yield of liquid products achieved using a Taizhuzgen zeolite catalyst with Mo concentration 2%. The gasoline fraction obtained using this catalyst mainly consisted of alkanes, aromatics and cycloalkanes, diesel fraction - alkanes, isoalkanes and cycloalkanes, heavy fraction – alkanes and aromatics. It is established that by modifying natural zeolite, Taizhuzgen with Mo salt affects the morphology of the catalyst and, as a result, affects the yield and composition of liquid fractions obtained from polymer waste in different ways.

Conflict of interest

All authors have read and are familiar with the content of the article and have no conflict of interest.

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