

## Activity features of catalysts for thermocatalytic hydrogenation processing of polymer waste

Zheneta Kh. Tashmukhambetova <sup>a</sup> , Tanakoz O. Kalamgali <sup>a</sup> ,  
Yermek A. Aubakirov <sup>a</sup> , Larissa R. Sassykova <sup>a\*</sup> ,  
Firuza Zh. Akhmetova <sup>b</sup> , Albina S. Alpysbay <sup>a</sup> 

a: Al-Farabi Kazakh National University, Almaty 050040, Kazakhstan

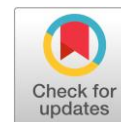
b: Zhangirkhan West-Kazakhstan Agrarian Technical University, Uralsk 090009, Kazakhstan

\* Corresponding author: [larissa.rav@mail.ru](mailto:larissa.rav@mail.ru)

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### Abstract

The aim of this study was to obtain new catalysts for the processing of carbon-containing polymer waste based on polyethylene and polypropylene, represented mostly by lids from beverages bottled in plastic containers, which accumulate in huge quantities in landfills, by the method of thermocatalytic hydrogenation into liquid fuels and other products. The process was carried out in the presence of fuel oil as a binder, a source of hydrogen and additional hydrocarbons. Thus, two tasks can be solved simultaneously: recycling the polymer waste and obtaining the alternative raw materials from the polymer waste in order to save resources and improve the environmental situation in general. New catalysts based on activated zeolite modified with Mo(VI) and W(VI) salts of various concentrations for the thermocatalytic hydrogenation processing of waste plastics into motor fuels were synthesized. The composition, structure, morphology and adsorption properties of the catalysts were determined by different physicochemical methods. The suitability of the obtained catalysts for use in the thermocatalytic hydrogenation processing of plastic waste into fuels was determined. The catalysts were tested during the processing of a mixture of polyethylene-polypropylene: a paste-forming agent (fuel oil) at  $T=450$  °C and a pressure of 0.6 MPa. The individual and group composition of gasoline, diesel and gas oil fractions was determined by chromatography coupled with mass spectrometry. The maximum yield of the gasoline fraction (16.9 wt.%) and diesel fraction (39.31 wt.%) was obtained on a 2%W(VI)/diatomite catalyst.

### Keywords

polymer waste  
thermocatalysis  
hydrogenation  
catalyst  
fuel  
diatomite

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### 1. Introduction

The problem of environmental pollution with carbon-containing industrial and household waste based on polymer and rubber products needs to be addressed. The main ways of the disposal of such waste are incineration and storage [1–15]. If we consider these carbon-containing wastes as an alternative source of hydrocarbons, then the development of new integrated technologies based on the use of effective catalysts will allow us to fully solve the existing environmental threats in the future. Moreover, it will make it possible to saturate the market with the necessary fuels, products and materials [16–23].

In the laboratory of the Department of Physical Chemistry, Catalysis and Petrochemistry of the Al-Farabi Kazakh National University, tests of new composite catalysts for the thermocatalytic hydrogenation processing of polymer waste were carried out. Composites based on Mo(VI) and W(VI) salts deposited on a diatomite substrate were studied for the first time as catalysts.

As is known, diatomite is a natural aluminosilicate material of a macroporous structure with a large internal surface formed as a result of the vital activity of organisms – diatomies. It has a complex “cemented” composition and contains inclusions of various minerals, such as silica, quartz, kaolin, opal, etc. Such a structure

allows us to consider it as a natural ion exchanger and adsorbent, as well as a substrate for the deposition of active catalysts [24–27].

The aim of this work was to synthesize new catalysts for the processing of carbon-containing polymeric wastes based on polyethylene and polypropylene, mainly represented by lids from drinks poured into plastic containers, which accumulate in large quantities in landfills, by thermal catalytic hydrogenation into liquid fuels and other products. The process was carried out in the presence of fuel oil as a binder, a source of hydrogen and additional hydrocarbons.

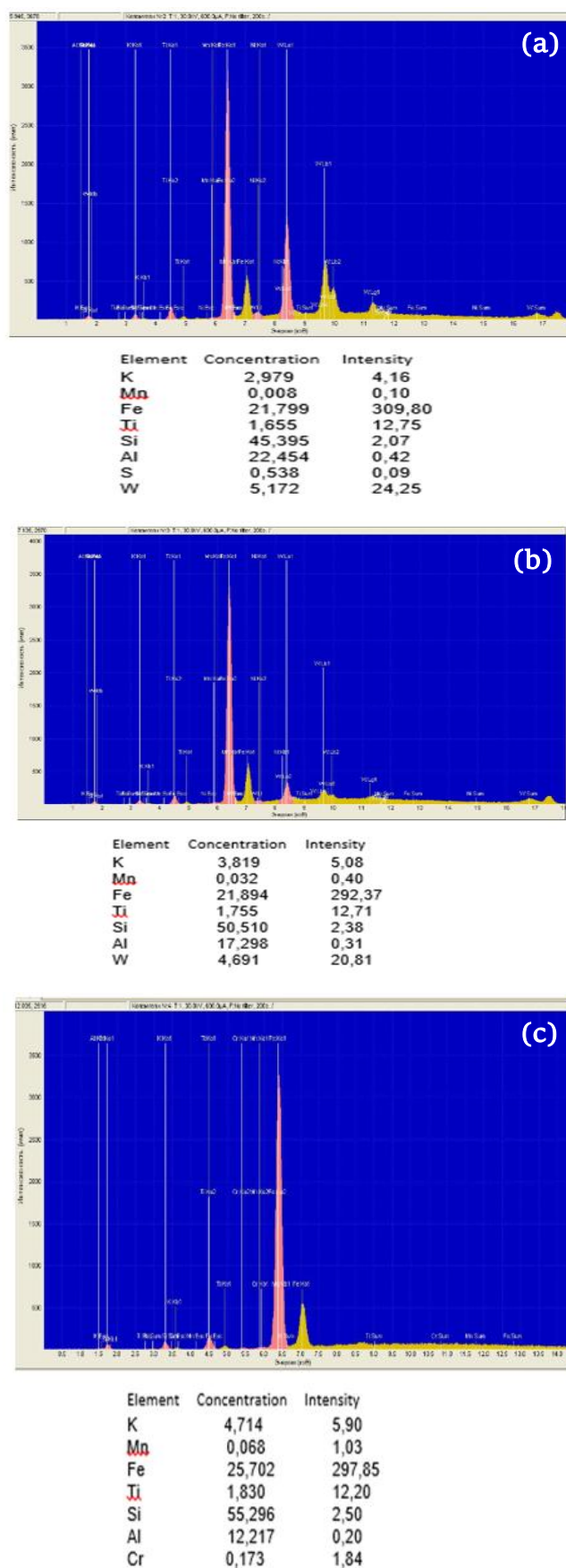
## 2. Experimental

In this work, diatomite from the Aktobe deposit of the Republic of Kazakhstan was studied. The concentration of the active metal in the composite was varied in the range from 1 to 2 wt.%. The effect of 4 different catalysts on the thermocatalytic hydrogenation processing of plastic waste was studied: diatomite activated by the acid-free method, 2% Mo(VI)/diatomite, 2% W(VI)/diatomite, and (1%Mo(VI) and 1%W(VI))/diatomaceous earth. As a feed-stock, polymeric wastes crushed to the state of crumbs from a waste processing plant in Almaty, represented by a mixture of polyethylene and polypropylene lids, were studied. To impart viscous paste-forming properties to the polymer mixture, fuel oil with a boiling point of more than 350 °C, obtained during the distillation processing of oil from the Kumkolskoe field, was used. As is known from our previous studies [6, 28–31], fuel oil was used not only as a paste-forming agent, but also as an additional source of hydrocarbons and hydrogen necessary for the hydrogenation reaction to proceed. In order to determine the composition, structure, morphology and adsorption properties of the studied catalysts, such physico-chemical methods of analysis as X-ray fluorescence, IR spectroscopy, Scanning Electron Microscopy (SEM), adsorption nitrogen porometry (BET), X-ray Diffraction analysis (XRD), thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTGA) were used.

## 3. Results and discussion

The presence of tungsten immobilized on the surface of diatomite during ion exchange in the composition of catalysts was established by X-ray fluorescence (Figure 1).

The process of ion exchange was accompanied by a decrease in the structure of diatomite of the concentrations of potassium, chromium, iron, as well as aluminum and silicon, which may indicate a possible destruction of the M–O–Si (where “M” is a metal) bond, a slight destruction of the Si–O–Si bond, and a partial removal of the six-coordinate aluminum.



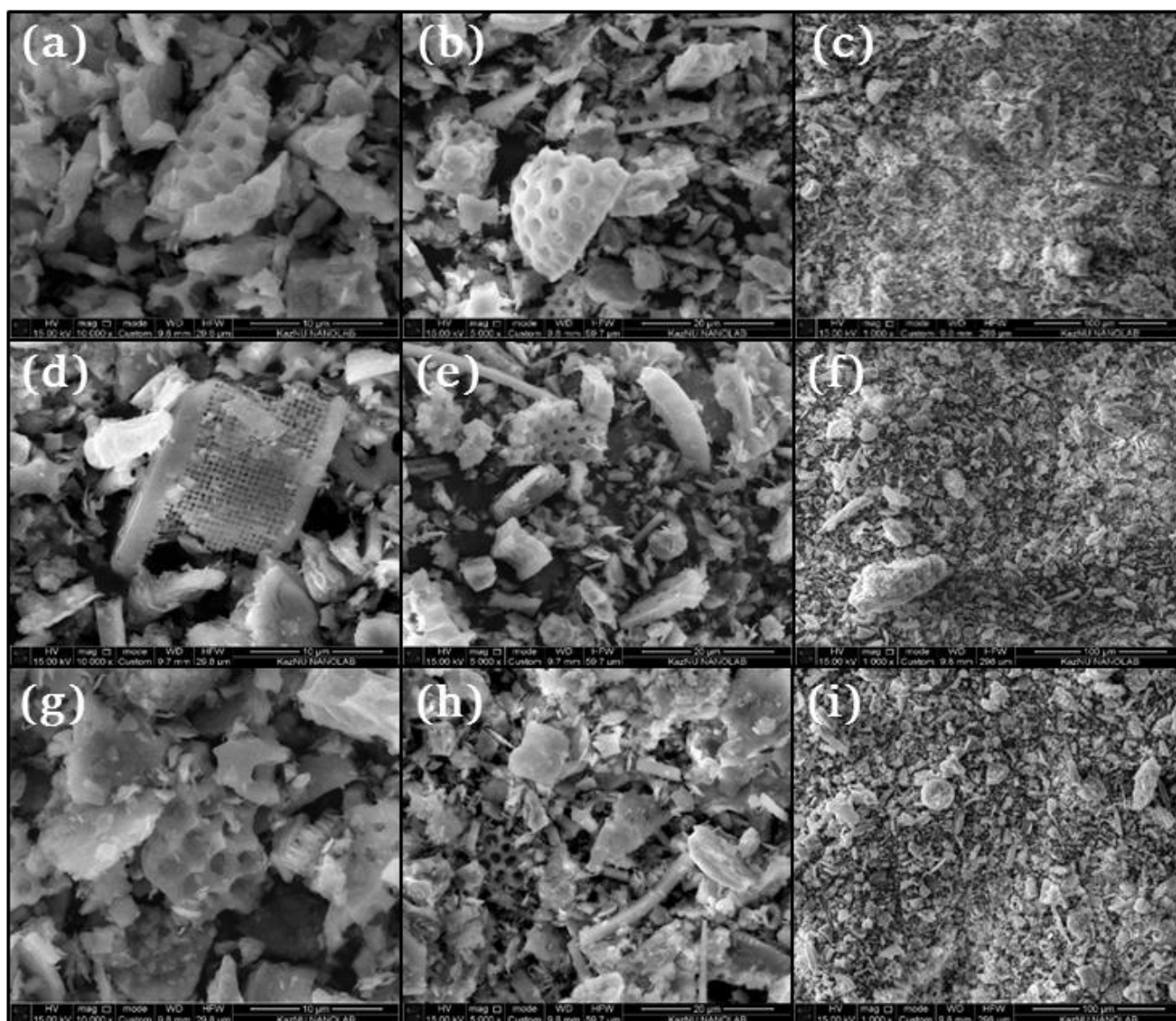
**Figure 1** Data of X-ray fluorescence analysis: 2% W(VI)/diatomite catalyst (a); the catalyst 1% Mo(VI) – 1% W(VI)/ diatomite (b); an activated diatomite (c).

So, for example, in the composition of activated diatomite, the concentration of Si was 65.30%, in 2% W(VI)/diatomite – 33.40%; in 2% Mo(VI)/diatomite – 45.40% in (1% Mo(VI) and 1% W(VI))/diatomite – 49.10%, respectively. The Al concentration was 12.22%, in 2% W(VI)/diatomite – 8.74%; in 2% Mo(VI)/diatomite – 22.45% in (1% Mo(VI) and 1% W(VI))/diatomite – 0%, respectively. The presence of molybdenum in the catalyst samples could not be determined by this method.

The study of the morphology of catalysts by SEM at different magnifications showed the presence in the images of distinct sections of the cellular structure inherent in diatomite, as well as loose and convex oval inclusions, most likely corresponding to the sites of destruction of diatomite and the introduction of molybdenum and tungsten ions into the substrate structure as a result of exchange with other ions (Figure 2).

The images clearly show integral fragments of the flaps of various organisms – diatoms with their inherent cellular structure, macropores with the inclusion of meso- and micropores, which indicates the heterogeneity of the surface of the diatomite. The samples of the studied catalysts, regardless of the content of the active metal, have a sufficiently developed specific surface area and are of interest for studying their structure by nitrogen porometry with a view to further use as a carrier of the active phase of the catalyst.

According to the data of the BET analysis (Table 1), in pure diatomite, the adsorption and desorption indices differ from those in the samples of catalysts with Mo and W active centers immobilized on them. This is also apparently due to a change in the structure of the catalyst after ion exchange treatment. The maximum value of the specific surface corresponds to the sample of 2% W(VI)/diatomite – 42.71 m<sup>2</sup>/g.



**Figure 2** Study of the morphology of the catalysts by SEM: 2% W(VI)/diatomite (10 μm (a), 20 μm (b), 100 μm (c)); 2% Mo(VI)/diatomite (10 μm (d), 20 μm (e), 100 μm (f)); 1% Mo(VI) – 1% W(VI)/diatomite (10 μm (g), 20 μm (h), 100 μm (i)).

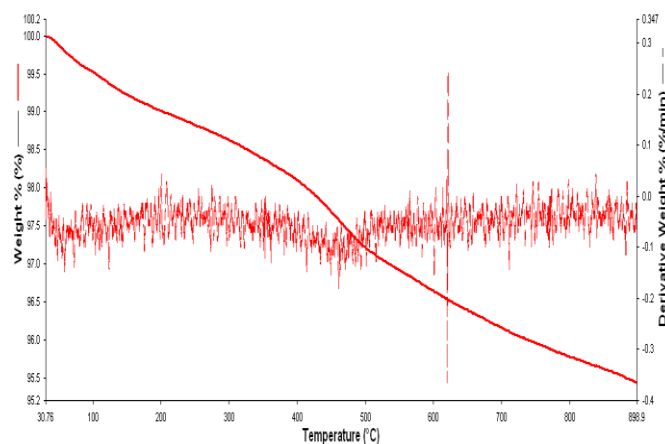


**Table 1** Determination of the specific surface area of catalysts by the BET method.

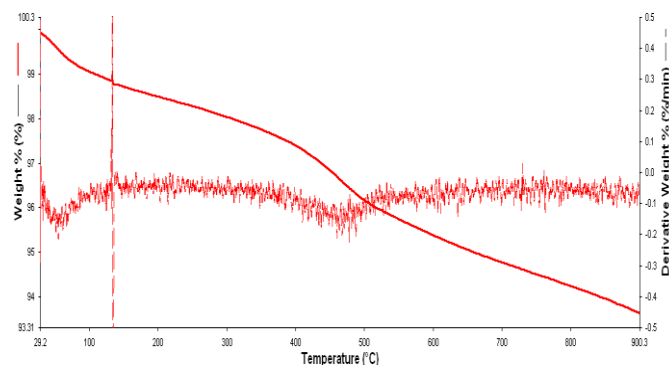
Catalyst	$S_{\text{specific surface area, m}^2/\text{g}}$
2% W(VI)/diatomite	60.76
2% Mo(VI)/diatomite	42.71
1% Mo(VI) - 1% W(VI)/diatomite	14.39
Activated diatomite	34.41

The study of catalyst samples by IR spectroscopy showed the presence in the spectra, mainly, of absorption bands characteristic of diatomite, since it has a complex composition and contains, in addition to Si and Al oxides, a number of oxides of various metals, such as Mn, Fe, Ti, Cr, K, etc. However, during the processing of Mo(VI) and W(VI) diatomite separately and with the total presence, a slight shift of the peak at  $1098.94 \text{ cm}^{-1}$  from 0.038 to  $0.045 \text{ cm}^{-1}$  is observed, corresponding to strong valence and deformation vibrations of Si-O-Si silica and quartz, as well as weak vibrations in the region of 460, 550, 804, 951  $\text{cm}^{-1}$ , valence fluctuations at 3450–3500  $\text{cm}^{-1}$ , corresponding to the OH group; valence fluctuations in the region of 3630–3695  $\text{cm}^{-1}$ , characteristic of clay and mica.

The activated diatomite and W(VI)/diatomite catalyst samples were analyzed by TGA and processed by DTGA. As the analyzes showed, the destruction of samples under the influence of temperature occurs intensively up to 130–135 °C, then it somewhat slows down until reaching 450–470 °C and then the final destruction occurs up to 898 °C (Figure 3 and 4). The TGA curves for both catalysts are approximately the same. The greatest mass loss of the sample, therefore, will be achieved already at the temperature of thermodestructive hydrogenation processing, which is 450 °C. However, the percentage of mass loss by the activated diatomite is insignificant and amounts to only 4.553%, and by the W(VI)/diatomite catalyst – 6.323%, respectively, which may indicate their resistance to temperature and the formation of disilicates (600–650 °C) with their further transition to the melt above 700 °C.

**Figure 3** Data of TGA and DTA analysis of activated diatomite.**Table 3** Chemical composition of the gasoline fraction ( $T_{\text{boiling point}} = 0\text{--}180 \text{ }^\circ\text{C}$ ) obtained on a 2% W/diatomite catalyst.

Liquid fraction	Hydrocarbons, %						
	Alkanes	Isoalkanes	Alkenes	Cycloalkanes	Cycloalkenes	Aromatic	Heterocompounds
0–180 °C	58.65	4.08	7.01	5.48	–	22.83	1.08

**Figure 4** Data of TGA and DTGA analysis of the catalyst W(VI)/diatomite.

Based on the X-ray phase analysis (XRD) of the studied catalysts, it was also found that the main contribution is made by the crystalline and amorphous phases of diatomite and minor fluctuations in intensity may be due to the presence of Mo(VI) and W(VI) salts.

The obtained catalysts were tested for processing a polyethylene-polypropylene mixture in the presence of a paste-forming agent at  $T = 450 \text{ }^\circ\text{C}$  and a pressure of 0.6 MPa. The maximum yield of the gasoline fraction ( $T_{\text{boiling point}} = 0\text{--}180 \text{ }^\circ\text{C}$ ) was observed on a catalyst of 2%W(VI)/diatomite – 16.90 wt. %.

The material balance of the most significant process under given conditions is presented in Table 2.

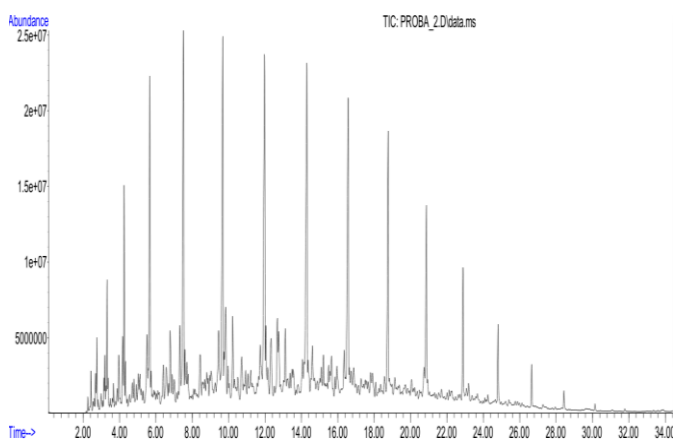
Table 3 shows the group composition of hydrocarbons of the gasoline fraction obtained on the catalyst 2% W/diatomite.

The hydrocarbon composition of distillates obtained on the synthesized catalysts was studied by chromatography-mass spectrometry. The chromatogram of the gasoline fraction boiling in the range of 0–180 °C obtained on a 2% W/diatomite catalyst is shown in Figure 5.

The main conclusions obtained as a result of the physicochemical studies and experimental tests of catalysts in this work are in agreement with the data mentioned in the scientific literature [2, 5, 32–41].

**Table 2** Material balance of the thermocatalytic hydrogenation processing of polymer waste on a catalyst 2% W(VI)/diatomite ( $T = 450 \text{ }^\circ\text{C}$ ,  $P = 0.6 \text{ MPa}$ ).

Taken	Wt.%	Consumption	Wt.%
Catalyst	2.00	Gasoline fraction 0–180 °C	16.90
Polymer waste	49.02	Diesel fraction 180–250 °C	39.31
		Heavy gas oil fraction 250–320 °C	–
Fuel oil	49.02	Losses, water,	30.08
		Gas	13.68
Total	100	Total	100



**Figure 5** Chromatogram of the gasoline fraction ( $T_{\text{boiling point}} = 0\text{--}180\text{ }^{\circ}\text{C}$ ) obtained on a 2% W(VI)/diatomite catalyst.

## 4. Conclusions

In this paper, the new composite catalysts based on activated zeolite modified with Mo(VI) and W(VI) salts of different concentrations for the thermocatalytic hydrogenation processing of plastic waste into motor fuels were developed. The basic physicochemical properties of the synthesized catalysts: the elemental and phase composition, surface morphology, specific surface area and the optimal destruction temperature were obtained. Based on the results of the experiments and the calculation of the material balance of the process, it was found that the catalyst 2% W/diatomite is the most active in the yield of the total liquid product. The effectiveness of the obtained composite catalysts is confirmed by the group hydrocarbon composition of fractions boiling up to  $180\text{ }^{\circ}\text{C}$ , from  $180$  to  $250\text{ }^{\circ}\text{C}$ , from  $250$  to  $320\text{ }^{\circ}\text{C}$ , determined by gas-liquid chromatography-mass spectrometry.

## Supplementary materials

No supplementary materials are available.

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## Author contributions

Conceptualization: Z.K.T.

Data curation: Z.K.T., L.R.S.

Formal Analysis: Y.A.A., L.R.S.

Funding acquisition: Y.A.A., Z.K.T.

Investigation: Z.K.T., T.O.K., A.S.A., F.Z.A.

Methodology: Z.K.T., Y.A.A.

Project administration: Y.A.A.

Resources: Z.K.T., Y.A.A., T.O.K.

Software: L.R.S.

Supervision: L.R.S., Z.K.T.

Validation: Z.K.T., L.R.S.

Visualization: Z.K.T., L.R.S., Y.A.A.

Writing – original draft: Z.K.T., L.R.S.

Writing – review & editing: Z.K.T., T.O.K., L.R.S.

## Conflict of interest

The authors declare no conflict of interest.

## Additional information

Author IDs:

Zheneta Kh. Tashmukhambetova, Scopus ID [56459076400](https://orcid.org/0000-0002-5645-9076);

Yermek A. Aubakirov, Scopus ID [55447002200](https://orcid.org/0000-0002-5544-7002);

Larissa R. Sassykova, Scopus ID [56178673800](https://orcid.org/0000-0001-5617-8673);

Firuzha Zh. Akhmetova, Scopus ID [57211321422](https://orcid.org/0000-0001-5721-1321).

Websites:

Al-Farabi Kazakh National University,  
<https://www.kaznu.kz/en/>;

Zhangirkhan West-Kazakhstan Agrarian Technical University,  
<https://wkau.kz/en/>.

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