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Y.K. Reimbayev[1] – *main author*,
U.G. Kanatbek[2], A.A. Kurtebayeva[3],
S.A. Orynbayev[4], T.G. Helder[5], M.S. Kalmakhanova[6]

[1,3]*PhD student*, [2]*Master student*, [4,6]*PhD, Associate Professor*, [5]*Professor*

ORCID
[1]*https://orcid.org/0009-0006-7669-3247* [2]*https://orcid.org/0009-0005-4568-9993*
[3]*https://orcid.org/0000-0002-1975-0755* [4]*https://orcid.org/0000-0002-5077-7219*
[5]*https://orcid.org/0000-0001-6898-2408* [6]*https://orcid.org/0000-0002-8635-463X*

[1,2,3,4,6]*M.Kh. Dulaty Taraz University, Taraz, Kazakhstan*
[5]*Centro de Investigação de Montanha (CIMO), Instituto Politécnico de Bragança, Bragança, Portugal*

[6]*marjanseitovna@mail.ru*

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APPLICATION OF NATURAL CLAY AND BIO-BASED ACTIVATED CARBON FOR THE ADSORPTIVE REMOVAL OF PARACETAMOL FROM WASTEWATER

Abstract. Pharmaceutical residues, particularly paracetamol, have emerged as persistent pollutants in aquatic environments due to their widespread use and incomplete removal by conventional wastewater treatment processes. This study investigates the potential of two low-cost and sustainable adsorbents – natural Kokshetau clay and activated carbon derived from peanut shells – for the removal of paracetamol from aqueous solutions. The adsorbents were characterized using XRD, and SEM, TEM, Elemental analyses to determine their surface properties and structural features. Batch adsorption experiments were conducted to evaluate the effects of pH, contact time, and initial concentration, while fixed-bed column studies were used to simulate continuous treatment conditions. The adsorption data were fitted to isotherm and kinetic models, with the Langmuir and pseudo-second-order models providing the best fit, respectively. Column performance was evaluated using the Thomas and Yoon-Nelson models. Results demonstrate that both materials exhibit high removal efficiency for paracetamol, with activated carbon showing superior performance. The findings highlight the potential of these materials as eco-friendly and effective adsorbents for pharmaceutical removal in water treatment applications.

Keywords: paracetamol, Kokshetau clay, activated carbon, peanut shell, pharmaceutical, adsorption, water treatment.

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Introduction. Pharmaceutical contaminants have emerged as significant environmental pollutants in modern water systems, primarily due to their widespread consumption and the inefficiency of conventional wastewater treatment technologies to fully eliminate them. Among these, paracetamol (acetaminophen) is frequently detected in surface waters, effluents, and even drinking water, often in concentrations ranging from nanograms to milligrams per liter. Its ubiquitous use

as an over-the-counter analgesic and antipyretic has contributed to its persistent input into aquatic environments. Although paracetamol is generally considered safe for human use, its presence in water bodies raises ecological concerns, particularly due to its bioactive nature and transformation into potentially toxic intermediates, such as 4-aminophenol [1-3].

Prolonged exposure to paracetamol residues and their byproducts has been shown to disrupt physiological, reproductive, and developmental functions in aquatic organisms, including fish, algae, and invertebrates. These impacts, along with the potential for bioaccumulation and the formation of hazardous secondary metabolites, underscore the need for effective, affordable, and sustainable methods for pharmaceutical removal from contaminated waters [4].

Conventional treatment methods, including advanced oxidation processes (AOPs), membrane filtration (e.g., reverse osmosis, nanofiltration), and ultraviolet irradiation, though effective under controlled conditions, are often cost-prohibitive, energy-intensive, and difficult to implement at full scale, especially in developing regions [5-7].

In response, there has been growing interest in the use of natural and waste-derived materials as adsorbents for water purification. Among these, natural clays and agricultural byproducts, stand out due to their wide availability, low cost, environmental compatibility, and favorable surface properties for adsorption. Activated carbon (AC) derived from peanut shells not only provides a sustainable method for waste valorization but also exhibits high surface area and porosity, making it an excellent adsorbent [8-10]. Similarly, naturally occurring clays, such as Kokshetau clay, possess layered structures and ion-exchange capacities that enhance their adsorption potential for various organic pollutants.

This study investigates the adsorption performance of raw Kokshetau clay and activated carbon produced from peanut shells for the removal of paracetamol from aqueous media. By conducting both batch and fixed-bed column experiments, and analyzing adsorption kinetics, isotherms, and material characteristics, the research aims to evaluate the feasibility of these materials as low-cost and eco-friendly alternatives for pharmaceutical pollutant remediation. The findings contribute to the broader effort of developing sustainable technologies for water treatment and environmental protection.

Materials and methods. A natural clay was collected from a clay deposit located in the city of Kokshetau, situated in the north of Kazakhstan. The raw clay was thoroughly washed with ultrapure water until a neutral pH was achieved, then dried in an oven at and sieved through a mesh.

Peanut shells, collected as agricultural waste, were similarly oven-dried, ground using a laboratory mill, and passed through a 100 μm sieve to obtain a fine and homogeneous powder. All materials were used without further chemical modification.

Preparation of Adsorbents. Kokshetau clay was obtained in its natural, raw form and subjected to purification by repeated washing with distilled water until the effluent reached a neutral pH (~6), ensuring the removal of soluble impurities and surface contaminants. The washed clay was subsequently dried in a muffle furnace at 60°C for 24 hours to eliminate residual moisture. Following thermal treatment, the dried material was ground and sieved through a 100 μm mesh to obtain a homogeneous particle size distribution suitable for use in adsorption studies.

Activated carbon (AC) was synthesized from peanut shells via a two-stage process consisting of carbonization and physical activation. Initially, the peanut

shells were washed with distilled water to remove adhering particulates, air-dried, and then ground and sieved to achieve uniform particle size. The carbonization step was conducted in a tubular furnace at a temperature range of 400-600°C for 1-2 hours under a nitrogen atmosphere to produce char. The charred material was subsequently activated in the same furnace by exposing it to a continuous flow of carbon dioxide or steam at elevated temperatures (800-900°C), thereby enhancing pore development and surface area. The resulting activated carbon was thoroughly washed with distilled water to remove residual ash and then dried at ambient temperature prior to use.

Experimental model.

Batch adsorption tests. The adsorption isotherm experiments of Kokshetau clay were conducted using aqueous paracetamol solutions with initial concentrations of 10, 25, 50, 75, and 100 mg/L. The pH of the solutions was maintained at approximately 6, reflecting the natural pH without adjustment. A fixed adsorbent dosage of 2.5 g/L of Kokshetau clay was used for all tests. The adsorption processes were carried out at room temperature (approximately 20-25°C) with a contact time of 24 hours to ensure equilibrium was achieved.

Adsorption kinetic experiments of Kokshetau clay were carried out using an initial paracetamol concentration of 10 mg/L in a total solution volume of 100 mL. An adsorbent dosage of 2.5 g/L was employed, and the suspensions were agitated using a magnetic stirrer at constant room temperature for a total contact time of 6 hours. At predetermined time intervals, aliquots were withdrawn, and the solid-liquid separation was performed by filtration through 0.45 μm hydrophilic nylon syringe filters to remove particulate matter. The residual concentration of paracetamol in the filtrate was subsequently analyzed using high-performance liquid chromatography (HPLC).

Adsorption isotherm experiments done with initial concentrations of paracetamol: 10, 25, 50, 75, 100, 150, 200, 250, 300, and 400 mg/L. The pH of all solutions was maintained at approximately 6 (natural pH) without adjustment. An adsorbent dosage of 2.5 g/L was used consistently throughout the experiments. Tests were conducted at ambient room temperature (approximately 20-25°C), with a contact time of 24 hours to ensure equilibrium was reached.

The adsorption kinetics of paracetamol onto activated carbon derived from peanut shells was investigated using an initial paracetamol concentration of 100 mg/L in 100 mL of aqueous solution. The adsorbent dosage was fixed at 2.5 g/L. The suspension was continuously agitated using a magnetic shaker at ambient temperature for a total duration of 8 hours. At specified time intervals, samples were withdrawn and immediately filtered through 0.45 μm hydrophilic nylon syringe filters to separate the adsorbent particles. The residual concentration of paracetamol in the filtrates was determined using high-performance liquid chromatography (HPLC).

Fixed bed column test. Fixed-bed column experiments were conducted to evaluate the dynamic adsorption performance of a binary adsorbent system composed of activated carbon derived from peanut shells and raw Kokshetau clay. A total of 0.5 g of adsorbent was used, consisting of 0.4 g of activated carbon and 0.1 g of clay. The adsorbent mixture was packed into a glass column following a standard layering protocol: a bottom layer of glass wool, followed by a layer of glass beads, the adsorbent bed, and an upper layer of glass beads to ensure uniform flow distribution and minimize channeling. A paracetamol solution with an initial concentration of 50 mg/L and natural pH of 6.0 was continuously passed through the column using an HPLC pump at a constant flow rate of 1 mL/min. Effluent

samples were collected at regular intervals and analyzed for residual paracetamol concentration using HPLC.

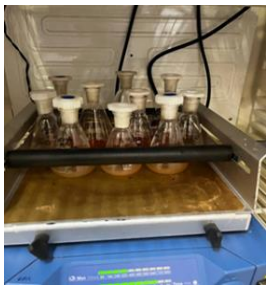


Fig. 1. Adsorption isotherm tests of clay and activated carbon

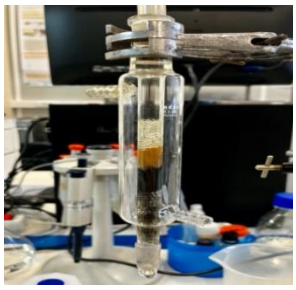

Fig. 2. Fixed Bed Column

Data analysis.

Isotherm and Kinetic models. The adsorption capacity of clay and activated carbon composites for paracetamol, expressed as the amount adsorbed per unit mass of adsorbent (mg/g), was evaluated, and the experimental data were fitted using non-linear forms of the pseudo-first-order, pseudo-second-order, and Elovich kinetic models, as represented by Equations (1)–(3), respectively.

$$qt = qe(1 - e- k1t) \quad (1)$$

$$t/qt = 1/k2q2\ e + t/qe \quad (2)$$

$$qt = 1/\beta\ *\ln(1 + \alpha\beta t) \quad (3)$$

The adsorption kinetics of PCM onto AC was studied over a 480-minute contact period using an initial PCM concentration of 100 mg/L. The experimental data were fitted to the pseudo-first-order (PFO), pseudo-second-order (PSO), and Elovich kinetic models (Fig. 3). The PSO model provided the best correlation with the experimental data, suggesting that the rate-limiting step may be chemisorption involving valence forces through sharing or exchange of electrons between adsorbent and adsorbate. Rapid adsorption was observed within the first 90 minutes, followed by a slower phase approaching equilibrium, with a maximum uptake of ~39.4 mg/g.

The equilibrium adsorption data obtained from the isotherm experiments were analyzed using the non-linear forms of the Langmuir, Freundlich, and Temkin isotherm models. These models are represented by Equations (4), (5), and (6), respectively.

$$q_e = q_m \times K_L \times C_e/1 + K_L \times C_e \quad (4)$$

$$q_e = K_F \times C^{\,1/n} \quad (5)$$

$$q\ e = B \cdot \ln (K\ T \cdot P) \quad (6)$$

The adsorption isotherm of paracetamol (PCM) onto activated carbon (AC) derived from peanut shells was investigated by varying the initial PCM concentration from 10 to 400 mg/L at a fixed adsorbent dosage (2.5 g/L) and pH 6. The adsorption equilibrium data were fitted using nonlinear Langmuir, Freundlich, and Temkin models (Figure 4). Among the models, the Langmuir isotherm

exhibited the best fit, indicating monolayer adsorption onto a homogeneous surface. The maximum adsorption capacity (q_max) was found to be approximately 232.1 mg/g. The high uptake at lower equilibrium concentrations (Ce < 10 mg/L) and the plateauing trend at higher Ce values confirm the saturation of active adsorption sites.

Breakthrough curves. Effluent concentration versus time (C–t) curves were analyzed to assess the performance of paracetamol removal under continuous flow conditions. The amounts of triclosan adsorbed at breakthrough (qb) and at saturation (qs), expressed in mg g^{-1}, were calculated using Equations (7) and (8), respectively:

$$q_b = (C_o \times Q / 1000 \times m) \times \int_o^{t_s} (1 - Cb/C_o) \quad (7)$$

$$q_b = (C_o \times Q / 1000 \times m) \times \int_o^{t_b} (1 - Cb/C_o) \quad (8)$$

where: C_o (mg L^{-1}) – the initial paracetamol concentration; Cb (mg L^{-1}) and Cs (mg L^{-1}) – the effluent concentrations at breakthrough and saturation, respectively; Q (mL min^{-1}) – the volumetric flow rate; m (g) – the mass of the CC/IO composite; tb and ts – the breakthrough and saturation times corresponding to C/C_o values of 0.1 and 0.9, respectively.

To further characterize the adsorption dynamics, the experimental data were also fitted to the non-linear form of the Yoon-Nelson model (Equation 9), which was selected for its simplicity and effectiveness in predicting breakthrough behavior and overall column performance.

$$C=Co/1+e^{TK-Kt} \quad (9)$$

A fixed-bed column experiment was conducted using a composite adsorbent composed of 0.4 g AC from peanut shells and 0.1 g raw Kokshetau clay. The influent paracetamol concentration was maintained at 50 mg/L, and the column was operated at pH 6.0 with a flow rate of 1 mL/min. The effluent concentration remained below detection ($C/C_o \approx 0$) up to 600 minutes, indicating high adsorption efficiency. Breakthrough began around 720 minutes, and saturation occurred approximately at 1320–1440 minutes, as evidenced by the gradual rise in C/C_o up to ~0.93. The slight pH increase in the effluent (from 6.0 to 7.2) may be attributed to surface exchange reactions or dissolution of basic sites on the adsorbent.The breakthrough data were fitted using nonlinear Yon-Nelson model (Fig. 5).

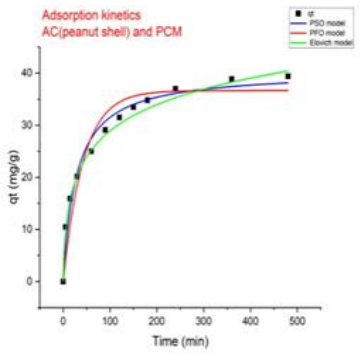

Fig. 3. PFO, PSO, Elovich kinetic models

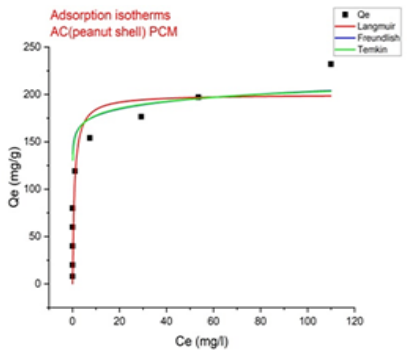

Fig. 4. Langmuir, Freundlish Temkin isotherm models

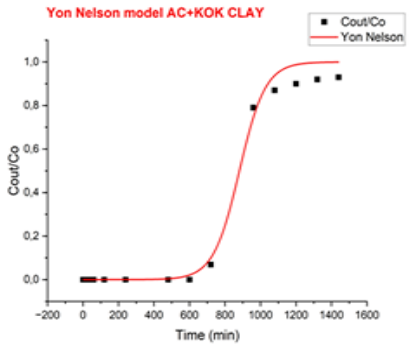

Fig. 5. Yon-Nelson model for fixed bed column

Research results and discussion. *Characterization of raw Kokshetau clay. X-ray Diffraction (XRD) Analysis*. The mineralogical composition of the raw Kokshetau clay was determined using X-ray diffraction (XRD). As shown in the diffractogram (Fig. 6), prominent reflections corresponding to muscovite, kaolinite, and quartz were identified. A notable peak at 2θ = 19.902° indicates the presence of muscovite, which is further confirmed by additional reflections in the 2θ range of 19.902-34.981° (d-spacing 4.457–2.376 Å). Kaolinite was identified by peaks in the 2θ range of 12.303-24.816°, and quartz was detected between 20.845-39.468° (Table 1).

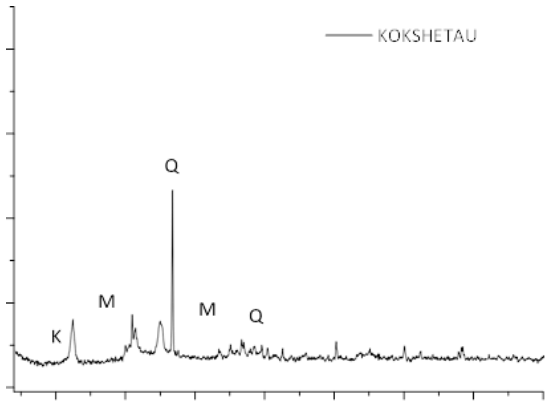

Fig. 6. XRD analysis of Kokshetau clay

Table 1

Reflections of the Kokshetau natural clays

№	Composition	deg. 2 θ	d (Å)
1	Kaolinite	12.303-24.816	7.188-3.584
2	Muscovite	19.902-34.981	4.457-2.376
3	Quartz	20.845-39.468	4.258-2.281

A semi-quantitative analysis based on the XRD spectra (Table 2) revealed that the Kokshetau clay is primarily composed of hydrated aluminum silicate, with muscovite constituting approximately 17.0%, and SiO_2 making up 28.3%. Additional phases include quartz and minor feldspar and calcite, indicating a heterogeneous natural composition with notable levels of mineral impurities. The presence of these impurities varies depending on the geological origin of the clay.

Table 2

Chemical composition of the Kokshetau clay determined by semi quantitative analysis from the XRD spectra

Mineral	Kokshetau
Quartz, SiO_2	28.3%
Calcite, $CaCO_3$	
Muscovite, $(K0.82Na0.18)\ (Fe0.03Al1.97)\ (AlSi_3)O_{10}(OH)_2$	17%
Albite endmember feldspar, $Na(AlSi_3O_8)$	1.6%
Hydrated aluminum silicate, $Al_2O_3 \cdot 4SiO_2 \cdot xH_2O$	0%
Kaolinite, $Al_2Si_2O_5(OH)_4$	53.1%
Microcline feldspar, KSi_3AlO_8	0%

Elemental Composition. The chemical composition of the raw Kokshetau clay was further analyzed using energy-dispersive X-ray spectroscopy (EDS) via an Inca Energy system (Table 3). The elemental analysis confirmed the presence of major constituents such as Al, Si, and O, consistent with aluminosilicate minerals. Trace elements including Na, Ca, and Fe were also detected, supporting the identification of muscovite and minor feldspar phases. The composition aligns well with the XRD findings and affirms the multi-phase nature of the natural clay.

Table 3

Chemical composition of the natural clays, determined by elemental analysis

Natural clay	Mass of the element (%)									
	O	Na	Mg	Al	Si	K	Ca	Ti	Mn	Fe
Kokshetau	54.71	*n.i.	0.15	13.4	19.17	0.28	0.21	1.43	n.i.*	10.66

*n.i. = not identified

Morphological Analysis (SEM and TEM). The surface morphology and microstructure of the raw Kokshetau clay were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) (Fig. 7 and 8). SEM images reveal a heterogeneous, layered morphology with irregular particle shapes and varying porosity, characteristic of natural clays. TEM analysis provided further insight into the particle arrangement at the nanoscale, showing layered silicate structures with interlamellar spacing, typical of muscovite and kaolinite clays. The porous and flaky texture is favorable for adsorption, offering a range of micro- and mesopores accessible to pollutant molecules.

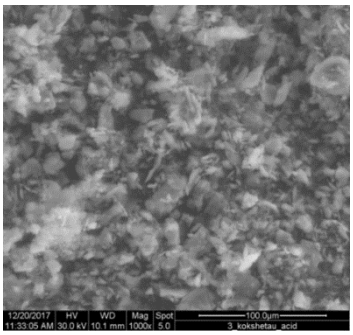

Fig. 7. SEM micrograph of Kokshetau clay

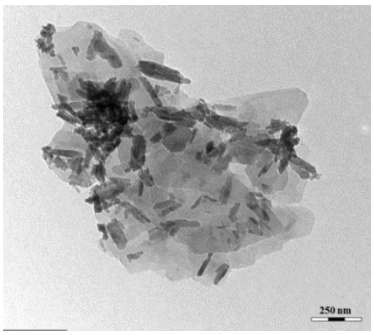

Fig. 8. TEM micrograph of Kokshetau clay

Conclusion. This study demonstrated the effective use of raw Kokshetau clay and activated carbon derived from peanut shells (AC-PS) as low-cost and environmentally friendly adsorbents for the removal of paracetamol (PCM) from aqueous solutions. Comprehensive characterization of the raw clay confirmed the presence of muscovite, kaolinite, and quartz, as well as a heterogeneous composition with layered morphology and microporous texture, which supports its adsorption capabilities.

Batch adsorption experiments revealed that AC-PS exhibited significantly higher adsorption capacity compared to raw clay, reaching a maximum of 232.06 mg/g. The adsorption equilibrium data fitted well with the Langmuir isotherm model, indicating monolayer adsorption on a homogeneous surface. Kinetic studies showed that the pseudo-second-order (PSO) model best described the adsorption process for both materials, suggesting chemisorption as the rate-limiting step.

The fixed-bed column experiments using a mixture of AC-PS and Kokshetau clay (4:1) further confirmed the high removal efficiency under dynamic flow conditions, with near-complete removal of PCM in the early stages of operation. These results demonstrate the potential of combining natural clay with low-cost biochar to design efficient and sustainable adsorption systems.

In conclusion, the synergistic use of natural clays and bio-derived activated carbon offers a promising approach for developing low-cost and efficient adsorbents for wastewater treatment applications, particularly for the removal of pharmaceutical contaminants like paracetamol.

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Е.Қ. Реимбаев[1], Ұ.Ғ. Қанатбек[1], А.А. Куртебаева[1], С.А. Орынбаев[1], T.G. Helder[2], М.С. Қалмаханова[1]

[1]М.Х. Дулати атындағы Тараз университеті, Тараз қ., Казақстан
[2]Браганса политехникалық институты, Браганса қ., Португалия

ТАБИҒИ САЗ ЖӘНЕ БИО-НЕГІЗДЕГІ АКТИВТЕЛГЕН КӨМІРДІ ДӘРІЛІК ҚАЛДЫҚ – ПАРАЦЕТАМОЛДЫ АҒЫН СУЛАРДАН АДСОРБЦИЯЛЫҚ ӘДІСПЕН ЖОЮҒА ҚОЛДАНУ

Аңдатпа. Фармацевтикалық қалдықтар, әсіресе парацетамол, өздерінің кеңінен қолданылуына және дәстүрлі ағын суларды тазарту әдістерінің оларды толық жоя алмауына байланысты су ортасында тұрақты ластаушылар ретінде қарастырылуда. Бұл зерттеуде парацетамолды су ерітінділерінен жою мақсатында төмен бағалы және экологиялық таза екі адсорбенттің – Көкшетау табиғи сазы мен жержаңғақ қабығынан алынған активтелген көмірдің – тиімділігі зерттелді. Адсорбенттердің беткі қасиеттері мен құрылымдық ерекшеліктерін анықтау үшін XRD, SEM, TEM және элементтік анализдер жүргізілді. Параметрлер ретінде pH, байланыс уақыты және бастапқы концентрацияны өзгерте отырып, сериялы адсорбциялық тәжірибелер жасалды, ал үздіксіз тазарту жағдайларын имитациялау үшін колонналық сынақтар жүргізілді. Адсорбция деректері изотермалық және кинетикалық модельдерге сәйкестендірілді, нәтижесінде сәйкесінше Лэнгмюр мен жалған екінші ретті модельдер жақсы сәйкестік көрсетті. Колонна жұмысы Томас және Юн-Нельсон модельдері арқылы бағаланды. Нәтижелер екі материалдың да парацетамолды тиімді жоятындығын, алайда активтелген көмірдің жоғары өнімділік көрсеткенін көрсетті. Бұл зерттеу көрсеткендей, аталған материалдар фармацевтикалық ластаушыларды жою үшін экологиялық қауіпсіз және тиімді адсорбенттер ретінде қолданылуға перспективалы.

Тірек сөздер: парацетамол, Көкшетау балшығы, активтендірілген көмір, жержаңғақ қабығы, фармацевтика, адсорбция, су тазарту.

Е.Қ. Реимбаев[1], Ұ.Ғ. Қанатбек[1], А.А. Куртебаева[1], С.А. Орынбаев[1], T.G. Helder[2], М.С. Қалмаханова[1]

[1]Таразский университет им.М.Х. Дулати, г. Тараз, Казахстан
[2]Политехнический институт Брагансы, г. Браганса, Португалия

ПРИМЕНЕНИЕ ПРИРОДНОЙ ГЛИНЫ И АКТИВИРОВАННОГО УГЛЯ БИОЛОГИЧЕСКОГО ПРОИСХОЖДЕНИЯ ДЛЯ АДСОРБЦИОННОГО УДАЛЕНИЯ ПАРАЦЕТАМОЛА ИЗ СТОЧНЫХ ВОД

Аннотация. Фармацевтические остатки, в частности парацетамол, становятся устойчивыми загрязнителями водной среды из-за их широкого применения и неполного удаления традиционными методами очистки сточных вод. В данном исследовании рассматривается потенциал двух недорогих и экологически безопасных адсорбентов – природной глины из Кокшетау и активированного угля, полученного из арахисовой шелухи – для удаления парацетамола из водных растворов. Адсорбенты были охарактеризованы с использованием методов XRD, SEM, TEM и элементного анализа с целью определения их поверхностных свойств и структурных характеристик. Пакетные адсорбционные эксперименты были проведены для оценки влияния pH, времени контакта и начальной концентрации, а также реализованы колонные исследования для имитации непрерывных условий очистки. Полученные данные были сопоставлены с изотермами и кинетическими моделями, причем наилучшее соответствие показали модели Лэнгмюра и псевдо-второго порядка соответственно. Эффективность колонн оценивалась с помощью моделей Томаса и Юн-Нельсона. Результаты показали высокую эффективность удаления парацетамола обоими материалами, при этом активированный уголь продемонстрировал более высокие показатели. Эти выводы подчеркивают потенциал указанных адсорбентов в качестве экологически безопасных и эффективных материалов для удаления фармацевтических загрязнителей в системах водоочистки.

Ключевые слова: парацетамол, кокшетауская глина, активированный уголь, арахисовая шелуха, фармацевтика, адсорбция, очистка воды.