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The influence of thermal and acid modification on the potential application of Jegłowa kaolin in cosmetic formulations

Introduction

Clay minerals constitute an important group of natural raw materials with broad application potential in the cosmetics industry (Viseras et al. 2021; Sarruf et al. 2024). Owing to their favorable sorption capacity, mineral composition, and ion-exchange properties, they are valuable components of cosmetic formulations intended for cleansing, soothing, and skin regeneration. Their low acquisition cost, wide availability, and environmentally benign character further enhance their attractiveness for industrial use (Guin et al. 2025). The application of clay minerals in skin care has a long-standing tradition, ranging from natural clay masks to modern dermatological formulations. At present, these materials are the subject of intensive research aimed at evaluating their physicochemical properties, biological safety, and functional performance in cosmetic products (Nkosi and Thembane 2024).

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The growing interest in mineral-based raw materials is driven not only by their natural origin and biocompatibility but also by increasing consumer demand for environmentally sustainable ingredients consistent with the "clean beauty" concept.

Among clay minerals, kaolin is the most extensively used in cosmetic applications. Due to its fine particle size, high purity, and surface reactivity, it functions as an active ingredient, emulsion stabilizer, or filler in a wide range of formulations. The role and applicability of clay raw materials in cosmetics have been comprehensively discussed in the literature (Carretero 2002; López-Galindo and Viseras 2004; Carretero and Pozo 2010; Mattioli et al. 2016; Viseras et al. 2021), where it is emphasized that the technological suitability and application performance of these materials are closely related to their physicochemical characteristics. The suitability of kaolin raw materials for cosmetic use is primarily determined by their chemical and mineralogical composition (Murray 1991; López-Galindo and Viseras 2004).

The dominant mineral phase in kaolin deposits is kaolinite, a 1 : 1 dioctahedral phyllosilicate with the chemical formula $Al_2Si_2O_5(OH)_4$. The crystal structure of kaolinite consists of alternating tetrahedral silica sheets and octahedral alumina sheets (Brindley and Robinson 1946; Bergaya and Lagaly 2013). The octahedral layer is mainly occupied by Al^{3+} cations; however, the very low net layer charge (close to zero) primarily results from the limited isomorphic substitution within the structure rather than the specific type of octahedral cation. Limited isomorphic substitutions by Fe^{3+} and Mg^{2+} ions may occur within the octahedral sheets (Meunier 2005). The individual layers are linked by hydrogen bonds of the Al–O–H···Si–O type, which contribute to the structural stability of kaolinite.

The stacking of structural layers may exhibit varying degrees of order, allowing the distinction of two main kaolinite varieties (Plançon et al. 1989). Tc-type kaolinite is characterized by a high degree of structural order and larger crystal size, whereas D-type kaolinite exhibits finer particle size and lower structural ordering. Disordered kaolinite typically displays higher plasticity compared to well-ordered Tc-type kaolinite (Lewicka et al. 2001). These structural differences influence surface reactivity and physicochemical behavior, thereby affecting the suitability of kaolinite for use in cosmetic formulations; for example, kaolinite Tc can be used in the preparation of organoclay composites for use as active ingredients in cosmetic formulations, whereas the low structural order of kaolinite D means that it is mainly used in peloid therapy due to its plastic properties. The physical and mechanical properties of kaolinite are closely related to its structural features. Minor substitutions of Fe^{3+} ions within the octahedral sheets contribute to a reduction in brightness and whiteness, parameters that are critical for cosmetic-grade kaolin.

The intrinsic properties of kaolin raw materials can be effectively optimized through appropriate processing and modification techniques, including the removal of abrasive impurities, thermal treatment, and acid activation. These procedures lead to the formation of modified materials with enhanced functional characteristics, such as increased sorption capacity, improved surface reactivity, and greater physicochemical stability under various environmental conditions (Yuan et al. 2013; Biswas et al. 2020; Singh 2022).

One of the key factors determining the suitability of kaolin for epidermal applications is its quartz content. The presence of quartz grains in cosmetic-grade raw materials is undesirable due to their abrasive nature and potential to cause microabrasions of the epidermis, which may compromise the integrity of the skin barrier (Mattioli et al. 2016). Moreover, excessive quartz content can adversely affect the homogeneity and rheological properties of cosmetic formulations. Therefore, clay raw materials intended for cosmetic use should be characterized by minimal or negligible quartz and other mineral impurities. The use of clay-rich raw material with a low quartz content is also advantageous in terms of minimizing processing costs, although quartz is relatively easy to remove from aqueous by washing in an aqueous environment.

To adapt the physicochemical properties of kaolin to the requirements of cosmetic formulations and to enhance its performance in contact with the skin, various modification methods are applied, including acid and thermal treatments. Acid activation, involving the treatment of kaolin with concentrated inorganic mineral acids, induces partial leaching of structural cations and the removal of associated mineral phases, particularly iron oxides and hydroxides. This process generates Lewis and Brønsted acid sites on the mineral surface, thereby enhancing its sorption capacity and chemical reactivity (Belver et al. 2002). Sorption capacity is a key parameter governing the functionality of mineral materials in cosmetic applications. Clay minerals, owing to their high specific surface area and developed pore structure, are capable of adsorbing water, sebum, pollutants, and organic compounds from the skin surface, making them effective components of cleansing and detoxifying formulations.

Thermal modification represents a crucial process aimed at enhancing the chemical reactivity of clay minerals through the removal of hydroxyl groups from their layered structures by controlled heating (Michot et al. 2011). This treatment induces a sequence of structural transformations that significantly influence the physicochemical behavior of the material. In the initial stage of thermal modification, occurring below 150°C, dehydration takes place, involving the removal of physically adsorbed water molecules bound to the mineral surface by van der Waals forces (Cheng et al. 2012). With increasing annealing temperature, dehydroxylation is initiated (Gasparini et al. 2013). This process typically occurs within the temperature range of 400–700°C, depending on the mineralogical composition of the raw material. During dehydroxylation, the rupture of O–H bonds within the octahedral sheets leads to the collapse of the layered structure, a decrease in crystallinity, and the formation of an amorphous phase with altered physicochemical properties (Kumari et al. 1986; Heller-Kallai 2006; Pandey et al. 2014; Cheng et al. 2019).

The aim of this study was to obtain thermally and chemically modified kaolin-based materials with enhanced functional properties and high microbiological quality suitable for cosmetic applications. Improving the functional performance of kaolin represents an important research direction, driven by the increasing demand for natural, safe, and biocompatible active ingredients in cosmetic products.

1. Materials and methods

1.1. The object of the study

The material investigated in this study was kaolin originating from the Jegłowa deposit, located in the Lower Silesia region, near Strzelin, within the metamorphic complex of the Strzelin Hills. The primary raw materials extracted from this deposit are quartzites and quartz–sericite schists. Kaolin, occurring as a secondary mineral phase, was formed as a result of hydrothermal alteration processes (Wyszomirski 1990). Within the deposit, it appears as an accessory mineral in the form of irregular lenses situated between layers of the main rock-forming components (Speczik et al. 2010). To date, there has been no documented industrial-scale extraction or utilization of kaolinitic raw material from these deposits.

1.2. Preparation of mineral material

To obtain the desired particle-size fraction, the raw kaolin sample was subjected to wet sieving using mesh sizes of 0.25 mm and 0.15 mm. Subsequently, sedimentation methods were applied to separate the clay fraction <0.063 mm. The prepared sample was then dried, manually ground in an agate mortar, and used for further modification (reference sample – KJ).

Four kaolin (in powder form) variants with modified surface characteristics were prepared as follows:

1. Thermally activated samples were obtained using a Nabertherm laboratory furnace at temperatures of 300°C (KJ300), 600°C (KJ600), and 900°C (KJ900). The heating rate was maintained at 100°C/h until the target temperature was reached, followed by a 3-hour holding period. The samples were subsequently cooled to ambient temperature under natural conditions.
2. The acid-activated sample was prepared by treatment with a 4 M hydrochloric acid solution (KJ4HCl). For modification, 220 cm^3 of HCl solution and 5 g of kaolin were introduced into a reaction flask. The mixture was heated using a thermostatically controlled heating mantle equipped with a magnetic stirrer and a temperature probe. A reflux condenser was used to prevent acid vapor loss. The sample was maintained at 75°C for 2 hours, then centrifuged (10 min, 3000 rpm) and repeatedly washed with deionized water. The washing procedure was repeated six times, after which a diagnostic test for chloride ions was performed using $AgNO_3$ solution, yielding a negative result.

All modification variants were prepared in triplicate.

1.3. Analytical methods

Particle Size Analysis by Laser Diffraction (PSA-LD)

Particle size analysis was carried out using aqueous suspensions of the samples on a Malvern Panalytical Master Sizer 3000 at the Institute of Ceramics and Building Materials in Warsaw.

X-ray diffraction (XRD) analysis

To determine the mineral composition of kaolin samples (in powder form) before and after modification, X-ray diffraction (XRD) analyses were performed. The measurements were carried out at the Laboratory of Electron Microscopy, Microanalysis, and X-ray Diffraction, at the Faculty of Geology, University of Warsaw, using a PANalytical X'Pert PRO MPD powder diffractometer (PANalytical B.V., The Netherlands) employing the Bragg–Brentano geometry. Data were collected over the 2θ range of 4–78°, with a step size of 0.026° 2θ and a time per step of 0.5 s. Powder samples were pressed and rotated at 1 rotation per second during measurement. The CoKα radiation (Fe filter) was applied under operating conditions of 30 mA and 40 kV. Diffraction patterns were recorded using a PIXcel fast linear detector. The total acquisition time per sample was approximately 4 hours.

Scanning electron microscopy (SEM)

Microscopic imaging of kaolin samples (in powder form) before and after modification was also performed at the same laboratory using a Zeiss VP scanning electron microscope (SEM). Imaging of powder samples was conducted using the SE2 secondary electron detector under low-voltage conditions (3 kV), with an aperture size of 20 μm and a working distance (WD) in the range between 8.4 and 8.7 mm. The samples were analyzed in bulk form without conductive coating.

Thermal analysis (TA)

Differential thermal analysis and thermogravimetric analysis (DTA–DTG) of the reference kaolin sample (in powder form) were conducted at the Laboratory of Applied Geology, at the Faculty of Geology, University of Warsaw, using an SDT Q600 thermal analyzer (TA Instruments). The heating rate was 10°C/min, and the measurements were performed up to 1000°C in an air atmosphere.

Gas porosimetry

Specific surface area (SSA) measurements were carried out at the Laboratory of Applied Geology, Faculty of Geology, University of Warsaw, using a gas adsorption analyzer ASAP 2020 (Micromeritics), with nitrogen (N_2) as the adsorbate at a constant temperature of –195°C. The analyses included the reference sample, acid-activated sample (KJ4HCl), and the thermally treated sample at 600°C (KJ600). Samples treated at 300°C and 900°C were not analyzed, as their results were expected to be comparable to those of the 600°C variant.

The specific surface area was determined using the Brunauer–Emmett–Teller (BET) method (eight-point analysis) and the Langmuir model. The pore size distribution was characterized using the Barrett–Joyner–Halenda (BJH) method and t-Plot analysis.

1.4. Microbiological quality assessment of mineral samples (quantitative and qualitative analysis)

Reference kaolinite samples and those subjected to modification were analyzed at the Laboratory of Geomicrobiology and Environmental Geochemistry, Faculty of Geology, University of Warsaw, to determine their microbiological quality. For this purpose, a kaolin paste was prepared by mixing kaolin with ultrapure (Barnstead, Easypure) and autoclaved water at a weight ratio of 1 part water to 3 parts kaolin, corresponding to a consistency similar to that of a cosmetic formulation.

The methodology of microbiological purity testing for cosmetic samples was based on the ISO 17516 standard, which includes both quantitative and qualitative analyses. The quantitative assessment comprised the determination of the total count of mesophilic aerobic bacteria, as well as yeasts and moulds.

The qualitative analyses focused on verifying the presence of specific potentially pathogenic microorganisms, including Gram-negative rods (*Escherichia coli*, *Pseudomonas aeruginosa*), Gram-positive bacteria (*Staphylococcus aureus*), and yeasts (*Candida albicans*).

The results obtained enabled the identification of kaolin samples that met the microbiological quality limits required for cosmetic products. The microbiological purity analyses performed in accordance with ISO 17516 included specific methodological standards, as summarized in Table 1.

The proliferation of microorganisms present in the tested mineral material was performed using Eugon Lecithin Broth. The total count of aerobic mesophilic bacteria was determined

Table 1. Criteria for the assessment of microbiological purity of mineral samples in accordance with ISO 17516

Tabela 1. Kryteria oceny czystości mikrobiologicznej próbek mineralnych zgodnie z normą ISO 17516

Microbiological purity testing (in accordance with ISO 17516)	Dedicated specific standards
Quantitative testing	Total Aerobic Mesophilic Microbial Count (PN-EN ISO 21149)
	Total Combined Yeast and Mould Count (PN-EN ISO 16212)
Qualitative testing	*Escherichia coli* (PN-EN ISO 21150)
	Staphylococcus aureus (PN-EN ISO 22718)
	Pseudomonas aureginosa (PN-EN ISO 22717)
	Candida albicans (PN-EN ISO 18416)

on tryptic soy agar (TSA), while Sabouraud dextrose agar (SDA) was used for the detection of fungi. Dey–Engley neutralizing broth was applied as a neutralizing agent. The cultures were incubated at 22.5 ± 2.5°C for 72 h. Colony counts for each dilution were performed in triplicate within the statistically significant range of 15–300 CFU per plate.

For qualitative analysis, selective and differential media were employed in accordance with ISO methodologies. MacConkey agar was used for the detection of *Escherichia coli*, Baird–Parker agar for *Staphylococcus aureus*, cetrimide agar for *Pseudomonas aeruginosa*, and Sabouraud agar supplemented with chloramphenicol for *Candida albicans* (media supplied by BioMaxima S.A.). All tests were conducted in triplicate. Incubation was carried out at 32.5 ± 2.5°C, and the presence of specific pathogenic microorganisms was evaluated after 48 h of incubation.

3. Results

Particle Size Analysis by Laser Diffraction (PSA-LD)

Particle size measurements were carried out on all samples following homogenization. In all samples tested, the proportion of particles smaller than 2 μm did not exceed 10% by volume. In the thermally modified samples, this proportion was significantly lower, which may be due to particle aggregation during sintering. The analysis of particle size measurements for thermally modified samples presents difficulties due to the processes mentioned above, which result in difficulties in the proper preparation of the suspension for testing and the presence of aggregates. The reference sample and the acid-modified sample exhibit similar median particle size values, D50 7.56 μm and 7.51 μm, respectively. In both samples, the fraction below 10 μm predominates. The results presented are consistent with the data reported by Speczik, Gasiński and Konopka 2010.

X-ray diffraction (XRD) analysis

To evaluate the effect of the applied modifications on the crystalline structure of kaolinite, X-ray diffraction patterns of acid-activated and thermally treated samples were compared with the reference kaolinite sample. The diffractogram of the unmodified kaolinite sample (KJ) revealed first-order reflections characteristic of kaolinite ($d = 7.19$ Å, $2\theta = 14°$), as well as a low-temperature quartz reflection of low intensity ($2\theta = 31°$, $d = 3.34$ Å), indicating the presence of a minor quartz admixture in the analyzed fraction. The sample annealed at 300°C (KJ300) (Figure 1) showed a minimal decrease in reflection intensity compared to the reference sample (KJ), while the positions of the diffraction peaks remained unchanged.

At higher annealing temperatures (600 and 900°C), a progressive decrease and eventual disappearance of kaolinite reflections were observed. The diffractogram of the sample treated at 600°C still exhibited a broad, low-intensity reflection at $d = 4.46$ Å ($2\theta = 23°$). At this temperature, the decomposition of kaolinite was not yet complete, and the weak reflections may be associated with residual short-range structural ordering within the material (Lee et al. 1999).

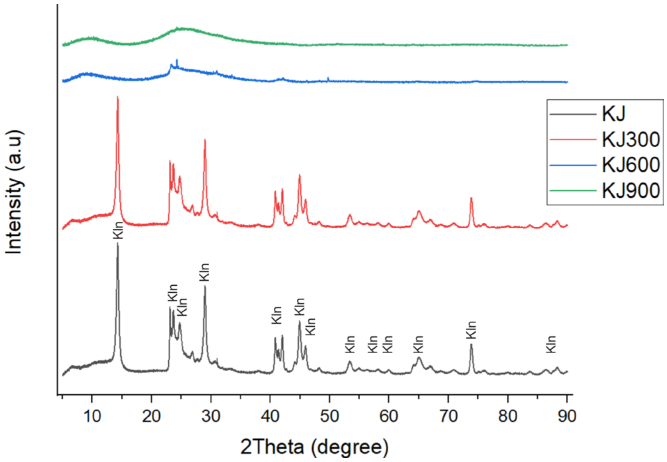

Fig. 1. X-ray diffractograms of thermally modified kaolin samples compared to reference sample

Rys. 1. Dyfraktogramy próbek modyfikowanych termicznie kaolinów w porównaniu z próbką referencyjną

At the highest activation temperature (900°C), no reflections corresponding to kaolinite were detected. The gradual decrease and eventual disappearance of kaolinite peaks are related to dehydroxylation processes and the breakdown of the kaolinite crystal structure, accompanied by the formation of amorphous metakaolinite phases (Brindley and Nakahira 1959; Ptáček et al. 2014). Increasing temperature induces thermal decomposition of kaolinite,

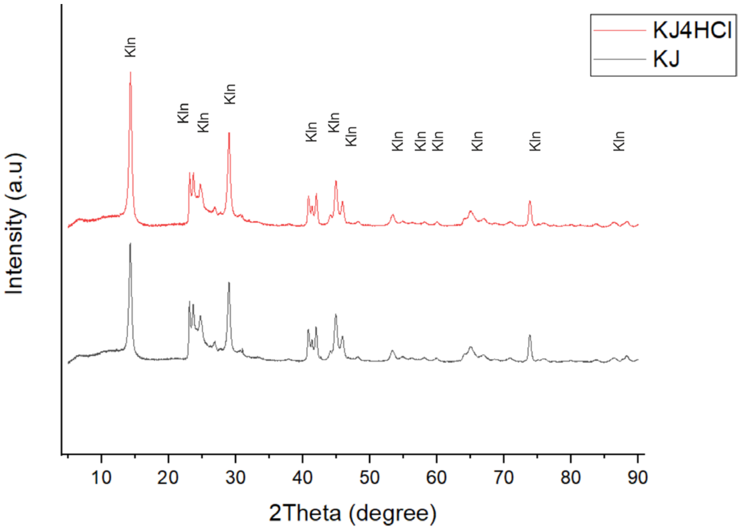

Fig. 2. X-ray diffractograms of acid-activated kaolin samples compared to reference sample

Rys. 2. Dyfraktogramy próbek kaolinów aktywowanych kwasowo w porównaniu z próbką referencyjną involving successive stages of dehydration and dehydroxylation that lead to structural collapse and metakaolinite formation (Brindley and Nakahira 1959). Further temperature increase promotes the development of new reactive mineral phases, such as spinels, which subsequently transform into mullite and cristobalite.

The diffractogram of the acid-activated sample in 4 M hydrochloric acid solution (Figure 2) is characterized by the presence of kaolinite reflections with increased intensity. The interplanar spacing of the first-order (001) kaolinite reflection is 7.19 Å. The observed shift in reflection position and the increase in their intensity are attributed to the removal of poorly crystalline mineral phases that may have been present as impurities in the reference sample. Additionally, this may indicate partial fragmentation of larger mineral platelets under the influence of acid treatment.

Scanning electron microscopy (SEM)

Observation of changes in grain size and surface morphology provides valuable insight into the effects of temperature and acid treatment on the mineral structure of the samples. Naturally occurring kaolin grains exhibit a typical hexagonal plate-like morphology (Figure 3a). These plates tend to form aggregates with a structure resembling pages in a book (Meunier 2005).

After thermal and acid activation, the shape of the plates is visibly altered, and the grain edges become serrated (Figure 3c, e). The size of the platelets varies across all samples, ranging from approximately 1 nm for the smallest to several or even more than a dozen nanometers for the largest.

In both thermally and acid-activated samples, the kaolinite grains form aggregates. However, in the case of the sample treated with hydrochloric acid (Figure 3c, e), the kaolinite plates are arranged in a more disordered manner compared to thermally activated samples (Figure 3b, d, f). The characteristic kaolinite "book-like" structure (Meunier 2005) is more distinctly preserved in the samples subjected to heat treatment. High hydrochloric acid concentration (4 M HCl) and increasing the annealing temperature (to 600°C and 900°C) result in greater dispersion of the aggregates (Figure 3d, e, f).

In the acid-activated sample, surfaces with diversified morphology were observed (Figure 3c, e), which can be attributed to the corrosive effect of hydrochloric acid on the surface of aggregated platelets, leading to the removal of Al^{3+} cations from the mineral structure (Komadel and Madejová 2006). A high acid concentration was observed to enhance the degradation of mineral platelets and surface structures.

Thermal analysis (TA)

Determining the temperature range at which the decomposition of the reference kaolin sample occurs enables the selection of appropriate temperature intervals for thermal modification. Thermal analysis confirmed the presence of kaolinite in the examined reference sample. The thermal decomposition of the mineral is presented graphically in the thermogram (Figure 4).

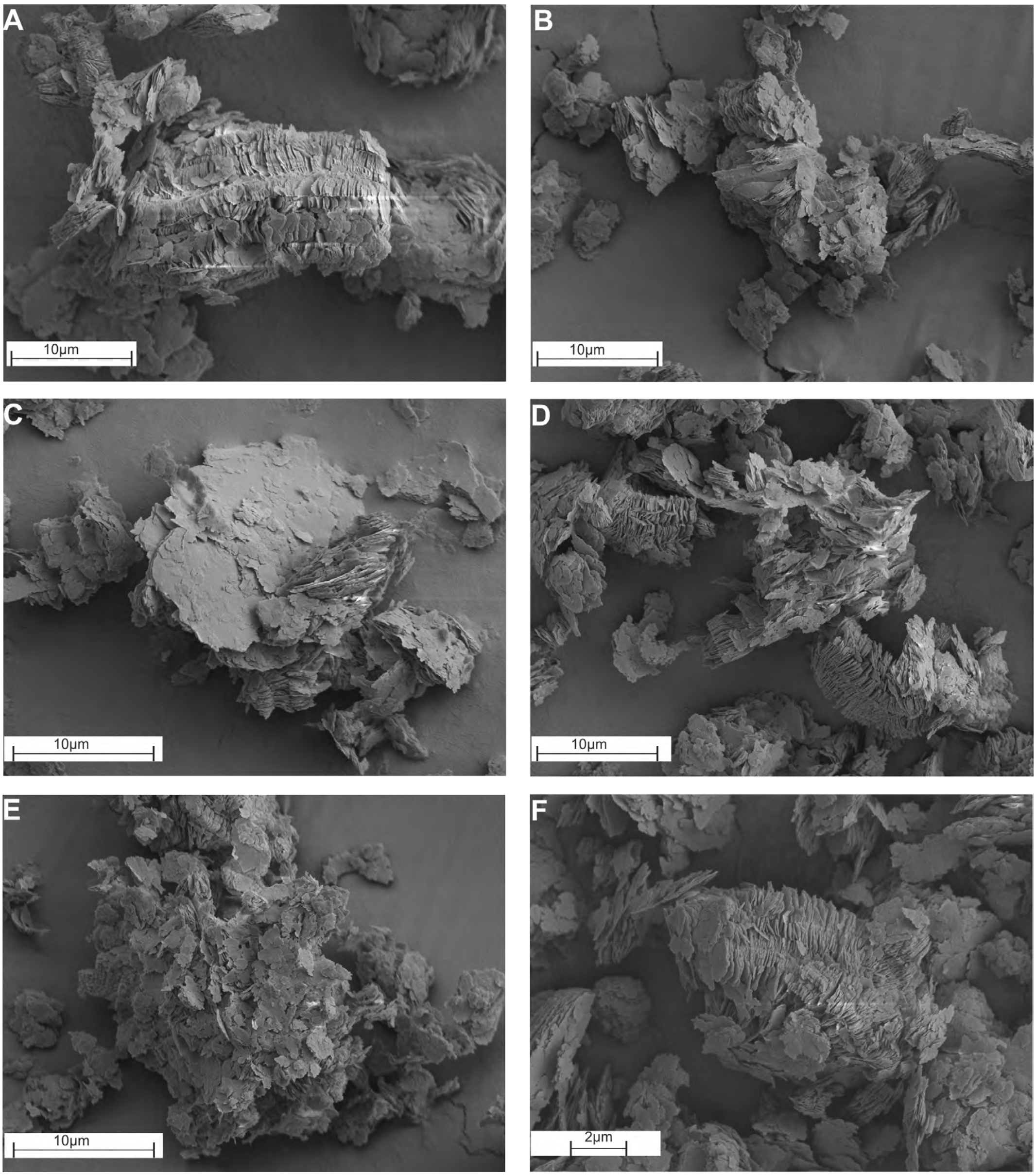

Fig. 3. SEM SE images
A – reference sample (KJ); B, D, F – thermally modified samples (B – KJ300°C, D – KJ600°C, F – KJ900°C); C, E – acid-activated sample (KJ4HCl)

Rys. 3. Fotografie SEM SE
A – próbka referencyjna (KJ); B, D, F próbki modyfikowane termicznie
(B – KJ300°C, D – KJ600°C, F – KJ900°C); C, E – próbka aktywowana kwasowo (KJ4HCl)

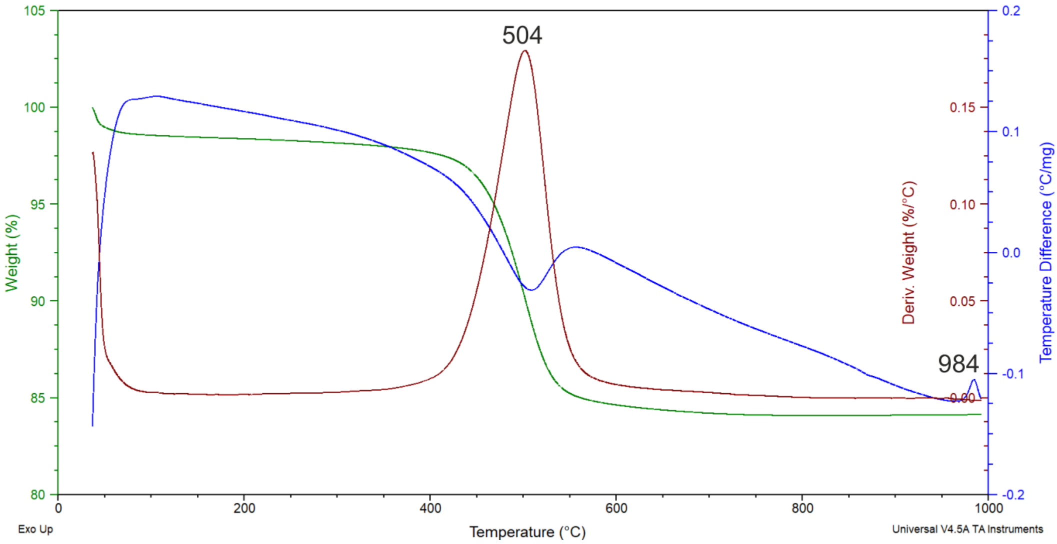

Fig. 4. Thermogram of kaolin KJ reference sample

Rys. 4. Termogram próbki referencyjnej KJ

An endothermic peak is observed at approximately 509°C, corresponding to the dehydroxylation of the kaolinite crystal lattice. This process occurs in two stages, involving the removal of both external and internal hydroxyl groups. The loss of structural water leads to the transformation of kaolinite into metakaolinite (Földvári 2011). The dehydroxylation reaction is accompanied by a mass loss of 13.59%, as shown on the TG curve.

At approximately 984°C, an exothermic peak was recorded, indicating phase transformations of kaolinite, including the formation of metakaolinite. The position and intensity of the exothermic peak are strongly influenced by the degree of the structural order of kaolinite, crystal size, and presence of impurities (Jasmund and Lagaly 1993; Földvári 2011). In the examined sample, the occurrence of the exothermic peak at a temperature below 1000°C can be attributed to the formation of a spinel-like phase.

Specific surface area analysis – gas porosimetry

The results of specific surface area measurements are presented in Table 2. The values obtained using the Langmuir method are higher than those determined by the BET method, which results from the different adsorption assumptions of these models. The specific surface area values determined by the BET method show variability among the tested samples.

The surface area of the reference sample was 14.87 m^2/g. In the thermally activated sample at 600°C, the surface area decreased to 12.10 m^2/g (a reduction of 18.6%), which can be attributed to kaolinite dehydroxylation and the partial collapse of the layered structure. In contrast, the sample treated with HCl exhibited significantly higher specific surface area values, reaching 34.35 m^2/g (an increase of 131%).

Table 2. Results of the measurement of adsorption parameters for reference kaolin and modified samples

Tabela 2. Wyniki pomiarów parametrów adsorpcyjnych badanych próbek kaolinu

Type of measurements	Samples		
	KJ	KJ600	KJ4HCl
BET	14.87 m^2/g	12.10 m^2/g	34.35 m^2/g
Single point	13.84 m^2/g	11.80 m^2/g	33.35 m^2/g
Langmuir	21.66 m^2/g	16.75 m^2/g	47.60 m^2/g
T-Plot (SSA of micropores)	–	1.85 m^2/g	4.59 m^2/g
BJH (average pore diameter)	164.34 Å	210.40 Å	104.40Å

The average pore diameter, determined using the BJH model, was 164.34 Å for the reference sample. In the sample heated to 600°C, this value increased by 28%, reaching 210.40 Å. For the acid-activated sample, the mean pore diameters decreased by 34.6%, reaching 104.40 Å, compared to the reference. According to the IUPAC pore classification (Thommes et al. 2015), samples with such pore characteristics are classified as mesoporous materials.

The micropore contribution to the total surface area was determined using the t-Plot method, which provided information on both the micropore surface area and volume. No micropores were detected in the reference sample. The lowest micropore content was found in the thermally modified sample, with a surface area of 1.85 m^2/g. In comparison, the 4 M acid-activated sample exhibited a surface area of 4.59 m^2/g.

Microbiological quality assessment of mineral samples (quantitative and qualitative analysis)

The mineralogical characterization of kaolinite was supplemented with microbiological quality analyses (based on ISO 17516) of both reference samples and those subjected to thermal and acid activation. These analyses were carried out to verify whether the applied clay mineral modification methods affect the microbiological safety of the resulting material. The quality of the tested mineral samples was assessed according to the acceptance criteria for the microbiological purity of cosmetic products specified in the same standard (Table 3).

The reference kaolin sample exhibited low microbiological quality. Only the application of modification procedures led to a significant improvement in this parameter. A significant increase in microbial growth was observed in the reference sample. The total number of mesophilic aerobic bacteria in this sample exceeded the microbiological limits permissible for cosmetic products (limit $\leq 1 \times 10^3$ CFU per g of product) (Table 3). Only the modified raw material samples, those calcined at 300, 600, and 900°C and acid-activated, were classified as microbiologically clean materials; in these cases, no bacterial or fungal growth was detected. No potentially pathogenic microorganisms, including *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, or *Candida albicans*, were detected in the reference sample or in the thermally and acid-modified samples.

Table 3. Acceptance criteria for the microbiological purity of cosmetic products and results of own research

Tabela 3. Akceptowane limity zanieczyszczeń mikrobiologicznych w próbkach kosmetycznych na podstawie ISO 17516 oraz wyniki badań własnych

Type of analysis	Criteria according to ISO 17516		Results of own research				
	Products (eye area, mucous membrane, children < 3 years old)	Other cosmetic products	KJ sample	Thermal modification			Acid modification
				KJ300	KJ600	KJ900	KJ4HCl
Total Aerobic Mesophilic Microbial Count	$\leq 1\times10^2$ CFU/g	$\leq 1\times10^3$ CFU/g	1.3×10^6 CFU/g	Absent	Absent	Absent	Absent
Total Combined Yeast and Mould Count	$\leq 1\times10^1$ CFU/g	$\leq 1\times10^2$ CFU/g	3.1×10^3 CFU/g	Absent	Absent	Absent	Absent
E.coli	Absence in 1 g or 1 ml	Absence in 1 g or 1 ml	Absent	Absent	Absent	Absent	Absent
S.aureus	Absence in 1 g or 1 ml	Absence in 1 g or 1 ml	Absent	Absent	Absent	Absent	Absent
P.aureginosa	Absence in 1 g or 1 ml	Absence in 1 g or 1 ml	Absent	Absent	Absent	Absent	Absent
C.albicans	Absence in 1 g or 1 ml	Absence in 1 g or 1 ml	Absent	Absent	Absent	Absent	Absent

CFU – Colony Forming Units; g or ml – per gram or per milliliter of the product; absent – not detectable in a 1g or 1ml of sample.

4. Discussion

Acid activation and thermal modification induce significant structural transformations within clay mineral samples (Nguetnkam et al. 2011; Cheng et al. 2012). The interaction of these activating factors with the mineral framework can lead to alterations in both mineralogical composition and sorption properties of the investigated materials (Sarma et al. 2011; Cheng et al. 2012). X-ray diffraction (XRD) analysis of thermally and acid-modified samples provides detailed insights into the magnitude and nature of structural changes occurring during these treatments (Panda et al. 2010; Gasparini et al. 2013).

In thermally treated samples, variations in the intensity of the first-order (001) kaolinite reflection indicate progressive structural decomposition with increasing treatment temperature (Sperinck et al. 2011). Diffractograms of the reference material and the sample heated at 300°C exhibit similar reflection intensities and line widths, suggesting that this temperature has a minimal impact on kaolinite structure. These subtle differences are primarily attributed to the loss of adsorbed water bound via van der Waals forces during dehydration (Cheng et al. 2012). At higher calcination temperatures (600–900°C), the disappearance of kaolinite (001) reflections reflects dehydroxylation and collapse of the layered structure, resulting in the formation of amorphous phases corresponding to metakaolinite (Gasparini et al. 2013; Cheng et al. 2019). Progressive amorphization at these temperatures yields a fine-grained material with enhanced mattifying properties, advantageous for cosmetic applications (Sarruf et al. 2025).

Acid activation using 4 M hydrochloric acid produces distinct structural effects, manifested by a reduction in the intensity of the primary (001) reflection and a slight shift toward higher 2θ angles compared to the reference sample (Sarma et al. 2011). According to Nguetnkam et al. (2011), acid treatment can induce bending of the layer-packet edges, increasing interlayer spacing (d-values). These acid-induced structural changes are functionally beneficial, as they enhance the accessibility of interlayer spaces to cosmetic formulation components, potentially promoting synergistic functional effects in final products.

Scanning electron microscopy (SEM) imaging allows for the comparative evaluation of microstructural changes induced by thermal and acid modifications. Acid-activated samples exhibit surface alterations on individual hexagonal platelets forming small kaolinite aggregates, indicating selective removal of entire layer packets (Alaba et al. 2015). This process is associated with the leaching of cations from octahedral and tetrahedral sheets, resulting in the formation of surface acidic sites (Nguetnkam et al. 2011). Thermally treated kaolinite shows reduced aggregate size and diminished lamellar dimensions, characteristic of thermal decomposition processes (Cheng et al. 2012). SEM observations complement XRD data, together providing a robust basis for interpreting the extent and nature of structural modifications.

Surface properties, including specific surface area (SSA), are strongly influenced by the type of modification. Samples treated with hydrochloric acid exhibit a marked increase in SSA. The factors influencing this enhancement, and consequently the sorption properties, include acid concentration, modification time, and sample type (Sarma et al. 2011).

The specific surface area of thermally modified samples at 600°C decreased relative to the reference, which can be attributed to structural transformations occurring during the calcination process (Cheng et al. 2012). However, Torres-Luna and Carriazo (2019) reported an increase in SSA for kaolin samples thermally treated above 700°C that had previously undergone acid activation. This effect results from complex reactions within the system, including the removal of mineral impurities during firing and the influence of the selected calcination parameters.

In the acid-activated sample, kaolinite platelets and aggregates undergo fragmentation, leading to the formation of pore spaces (Alaba et al. 2015). These processes contribute to the overall increase in specific surface area. During activation at temperatures between 450–660°C, dehydroxylation occurs, which induces collapse of the layered structure and consequently reduces SSA due to densification of the material framework.

Fragmentation of kaolinite platelets during acid activation generates micropores, enhancing sorption capacity. The t-Plot method revealed that micropores were absent in the reference Jegłowa kaolin but appeared after surface modification. Acid-activated samples exhibited higher micropore volume and surface area compared to thermally treated material at 600°C, indicating that chemical activation exerts a stronger influence on microporosity than thermal treatment (Alaba et al. 2015). The leaching of Al^{3+} cations during acid activation contributes to the formation of acidic sites and increased sorption potential (Nguetnkam et al. 2011). Acid activation of a kaolin sample from Jegłowa increases its specific surface area and, consequently, improves the sorption properties of the sample. As a result, the modified mineral raw material can be used not only in face masks for normal skin or creams (Sarruf et al. 2024) but also in dermocosmetic formulations, which is linked to the formation of Lewis and Brønsted acid centres (Christidis et al. 1997) as a result of the action of concentrated inorganic acid. The presence of these centers enables more effective removal of sebum-constituting compounds and skin metabolite residues.

Thermal modification, including dehydroxylation and metakaolinite formation, negatively affects microporous surface development. Consequently, thermal and chemical activation methods yield materials with divergent functional properties suitable for distinct cosmetic applications. Thermally activated kaolins produce fine-grained particles suitable as fillers in white cosmetics or as mattifying agents in colored formulations. In contrast, acid-activated kaolins, with enhanced sorption capacity, are highly suitable as active ingredients in astringent and cleansing cosmetic products, including dermocosmetic applications.

The microbiological quality testing of cosmetics and raw materials intended for cosmetic production is an essential element of product safety verification. Each modification method and the resulting alterations in mineral structure markedly affected the microbiological quality of the test material. Based on the results, only the samples of materials subjected to modification processes met the microbiological limit criteria defined in the international standard ISO/DIS 17516 for cosmetics. Calcination of kaolin at 300°C not only completely eliminates microorganisms but also removes residual organic matter and partially modifies the mineral structure. Such a high temperature leads to the oxidation and denaturation of proteins as well as the destruction of lipid membranes (Russell 2003), and with an adequate exposure time, results in complete sterilization of the material.

In the case of acid modification, the presence of hydrochloric acid causes immediate protonation of amino, amide, and carboxyl groups in microbial proteins, disrupting the hydrogen and ionic bonds that stabilize their secondary and tertiary structures. Additionally, the acidification of the cell interior leads to depurination and depyrimidination of DNA as well as RNA degradation (Foster 2004; Mols and Abee 2011). Both modification processes, thermal and acid, result in the complete elimination of microorganisms.

Conclusions

The results of the conducted studies allow the following conclusions to be drawn:

1. The thermally modified sample treated at a temperature of 600°C exhibited an altered mineral composition, with a dominant presence of amorphous metakaolinite forms. This treatment resulted in a reduction of the specific surface area of kaolin, which may suggest a potential decrease in sorption capacity. Although complete microbiological safety was achieved, this effect may limit the applicability of the material in cosmetic formulations.
2. Acid-activated kaolin was characterized by a significant increase in sorption parameters, micropore volume, and the density of acid sites, while preserving the crystalline structure of kaolinite and maintaining high microbiological quality. These features enhance the material's potential for use in cosmetic products.
3. Materials obtained through acid activation, exhibiting enhanced sorption properties, demonstrate high potential for practical application as functional mineral raw materials in astringent and cleansing cosmetic formulations, including dermocosmetic products.
4. Unmodified kaolinitic raw materials can achieve microbiological safety suitable for cosmetic applications only when combined with an appropriate antimicrobial agent compatible with natural cosmetic formulations.

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THE INFLUENCE OF THERMAL AND ACID MODIFICATION ON THE POTENTIAL APPLICATION OF JEGŁOWA KAOLIN IN COSMETIC FORMULATIONS

Keywords

Jegłowa kaolin, cosmetic application of clays, acid activation,
thermal modification, microbiological quality

Abstract

The aim of this study was to develop a clay-based mineral material with enhanced sorption properties suitable for application as an active ingredient in cosmetic formulations. Kaolin samples from the Jegłowa deposit were subjected to structural modifications through thermal treatment at 300, 600, and 900°C, as well as acid activation using a 4 M HCl solution. The resulting materials were assessed for microbiological quality in accordance with the ISO/DIS 17516 standard. In addition, mineralogical composition (X-ray diffraction and thermal analysis), specific surface area, pore structure, and sorption properties (gas porosimetry, SEM) were characterized to evaluate the influence of the type and intensity of modification on the functional properties of kaolin.

Thermal modification at 600°C induced decomposition processes, resulting in a reduction in structural order and a decrease in specific surface area, which consequently limited the material's sorption capacity. In contrast, acid activation led to a significant increase in the specific surface area of kaolin, which is advantageous for sorption processes occurring at the interface between the modified clay material and the epidermal layers. Microbiological analyses revealed that unmodified kaolin samples did not meet the requirements specified in the PN-EN ISO 17516 standard.

Satisfactory microbiological quality was achieved exclusively in samples subjected to combined thermal and acid modification. Based on these results, it was concluded that unmodified Jegłowa kaolin can be incorporated into cosmetic formulations only in combination with antimicrobial agents to ensure compliance with microbiological quality requirements of the final product.

WPŁYW MODYFIKACJI TERMICZNEJ I AKTYWACJI KWASOWEJ KAOLINU Z JEGŁOWEJ NA MOŻLIWOŚCI APLIKACJI W PRZEMYŚLE KOSMETYCZNYM

Słowa kluczowe

kaolin z Jegłowa, surowce ilaste w kosmetyce, aktywacja kwasowa, modyfikacja termiczna, jakość mikrobiologiczna

Streszczenie

Celem badań było otrzymanie mineralnego materiału ilastego o podwyższonych właściwościach sorpcyjnych, umożliwiających jego zastosowanie jako składnika aktywnego w formulacjach kosmetycznych. Próbki kaolinu pochodzące ze złoża Jegłowa zostały poddane modyfikacjom strukturalnym obejmującym: modyfikację termiczną w temperaturach 300°C, 600°C i 900°C oraz aktywację kwasową z zastosowaniem 4 M HCl. Uzyskane materiały poddano ocenie czystości mikrobiologicznej zgodnie z wymaganiami międzynarodowej normy ISO/DIS 17516. Określono zmiany w składzie mineralnym (XRD, TA), powierzchni właściwej, strukturze porowatej oraz potencjale sorpcyjnym (porozymetria gazowa, SEM), co pozwoliło na ocenę wpływu rodzaju modyfikacji na właściwości funkcjonalne kaolinu. Modyfikacja termiczna w 600°C spowodowała procesy dekompozycji termicznej. Zmiany te skutkowały redukcją uporządkowania strukturalnego minerału oraz zmniejszeniem powierzchni właściwej, co ograniczało jego potencjał sorpcyjny. Aktywacja kwasowa doprowadziła do znacznego zwiększenia powierzchni właściwej kaolinu, co jest korzystne dla procesów sorpcyjnych zachodzących w kontakcie epidermy ze zmodyfikowanym surowcem ilastym.

Wyniki analizy czystości mikrobiologicznej wykazały, że próbki kaolinu niemodyfikowanego nie spełniały wymagań określonych w normie PN-EN ISO 17516. Wysoki poziom czystości mikrobiologicznej uzyskano jedynie w przypadku próbek materiału modyfikowanego, zarówno termicznie, jak i kwasowo. Na tej podstawie stwierdzono, że kaolin Jegłowa w postaci niemodyfikowanej może być stosowany w formulacjach kosmetycznych wyłącznie w połączeniu ze środkami przeciwdrobnoustrojowymi, które zapewniają spełnienie wymagań dotyczących jakości mikrobiologicznej produktu końcowego.