

The Effect of Inorganic Acid Activation on the Metal Oxide Content of Natural Montmorillonite from Gowa Regency

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ABSTRACT

Natural montmorillonite has attracted considerable attention due to its high cation exchange capacity, layered structure, and potential applications as adsorbents and catalyst supports. However, it generally contains impurity oxides that affect its physicochemical properties. This study aimed to investigate the effect of inorganic acid activation on the metal oxide content and crystalline characteristics of natural montmorillonite obtained from Gowa Regency, South Sulawesi, Indonesia. The montmorillonite samples were activated using 2 M HCl and 2 M H₂SO₄ through acid leaching treatment. The metal oxide composition and crystalline phases were characterized using X-ray fluorescence (XRF) and X-ray diffraction (XRD), respectively. The XRF results revealed that Fe₂O₃ was the dominant impurity oxide in natural montmorillonite with a concentration of 31.61%. After acid activation, the Fe₂O₃ content decreased significantly to 21.78% for HCl-treated MMT and 18.83% for H₂SO₄-treated MMT, indicating effective removal of iron oxide impurities. In contrast, the SiO₂ content increased from 39.51% to 40.13% and 41.02% after HCl and H₂SO₄ activation, respectively. XRD analysis confirmed the presence of quartz silica, alumina, and hematite phases, with diffraction peaks at $2\theta \approx 26.6^\circ$, 34.2° , and 50.1° , respectively. Acid activation also reduced the average crystallite size from 41.2 nm in natural MMT to 35.1 nm and 29.9 nm after HCl and H₂SO₄ treatment, respectively. The results demonstrate that inorganic acid activation effectively modified the oxide composition and crystalline structure of natural montmorillonite, particularly through the removal of hematite impurities and enhancement of silica-rich phases. Therefore, acid-activated montmorillonite shows promising potential for adsorption and catalytic applications.

KEYWORDS

Acid leaching, montmorillonite, metal oxide

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A. Introduction

Natural clay minerals, particularly montmorillonite, have attracted significant attention due to their abundant availability, layered structure, high cation exchange capacity, and wide range of industrial applications. Montmorillonite is commonly utilized as an adsorbent, catalyst support, and precursor material in environmental remediation because of its unique physicochemical properties (Ria et al., 2025). However, the natural form of montmorillonite generally contains impurities and metal oxides that influence its adsorption performance and structural characteristics (Rahman et al., 2023). Therefore, modification through acid activation has become one of the most effective methods to improve the properties of natural montmorillonite (Side et al., 2023).

Acid activation using inorganic acids can alter the mineral structure and chemical composition of montmorillonite by removing exchangeable cations and dissolving certain metal oxides from the clay framework. Several studies have reported that acid treatment significantly affects the content of

oxides such as SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , and CaO ([Side et al., 2023](#); [Side & Putri, 2022](#)). For instance, activation using hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) was found to increase silica content while reducing aluminum and iron oxides due to dealumination processes ([Katsuki et al., 2026](#)). Previous research also demonstrated that stronger inorganic acids tend to produce greater structural modification and surface area enhancement compared to weaker acids ([Ma et al., 2026](#)). These modifications are highly important because the oxide composition directly influences the adsorption capacity, catalytic activity, and thermal stability of montmorillonite (Ria et al., 2025).

The previous study reported that natural clay from Gowa Regency possesses significant montmorillonite characteristics with considerable metal oxide content, especially silica and alumina compounds ([Bejar et al., 2016](#); [P. Zhou et al., 2024](#)). The study revealed that local clay resources from Gowa have promising potential for further development as low-cost adsorbent materials and environmentally friendly catalysts ([Putri et al., 2024, 2025](#); [Side et al., 2024](#)). Nevertheless, the previous study mainly focused on the characterization of natural clay without extensive investigation regarding the influence of different inorganic acids on changes in metal oxide composition.

Compared with previous studies, the present research emphasizes the comparative effect of several inorganic acids on the metal oxide content of natural montmorillonite obtained from Gowa Regency, South Sulawesi. This study is important because different acids may produce distinct dissolution mechanisms and structural modifications within the clay mineral. Furthermore, local montmorillonite resources from Gowa Regency remain relatively underexplored despite their potential industrial value. By evaluating the changes in oxide composition after acid activation, this research is expected to provide scientific information regarding the most effective inorganic acid treatment for improving the characteristics of natural montmorillonite.

Therefore, this study aims to analyze the effect of inorganic acid activation on the metal oxide content of natural montmorillonite from Gowa Regency. The findings of this research are expected to contribute to the development of modified clay materials for adsorption, catalysis, and other industrial applications based on local natural resources.

B. Method

Preparation of natural montmorillonite

The natural montmorillonite (MMT) used in this study was obtained from Pallangga sub-district, Gowa Regency of South Sulawesi Province of Indonesia. Natural MMT sample was first homogenized and subsequently dried under sunlight to reduce its moisture content. The dried sample was then crushed and sieved through a 200-mesh sieve, and only the particles passing through the sieve were used for further analysis. The natural montmorillonite samples were subsequently characterized to determine their silica–alumina content and crystalline phases using XRF and XRD analyses. The illustration of montmorillonite preparation is presented in Figure 1.

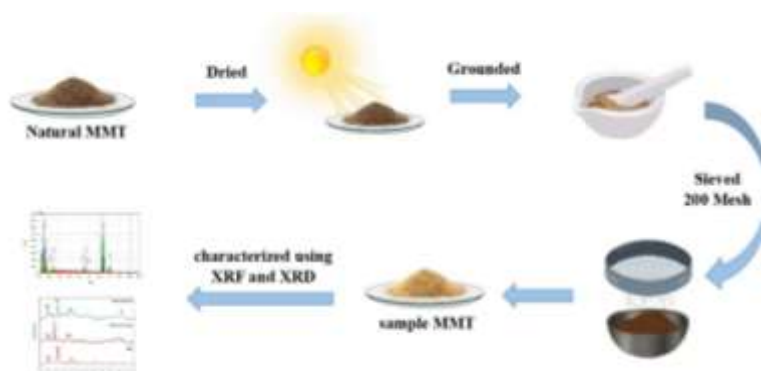


Figure 1. Illustration of natural MMT preparation

Activation of natural montmorillonite

A total of 100 g of prepared natural montmorillonite was placed into a beaker glass, followed by the addition of 1000 mL of distilled water while being continuously stirred using a magnetic stirrer for approximately 3–4 hours. The montmorillonite was subsequently filtered and dried at 50 °C until a constant weight was obtained. Thereafter, 100 mL of 2 M HCl and H₂SO₄ solutions were added separately to 100 g of the washed montmorillonite and stirred for 14–16 hours using a magnetic stirrer. The activated montmorillonite was then washed repeatedly with distilled water until free from residual acid and dried at 120 °C until reaching a constant weight. The dried sample was further ground and sieved through a 200-mesh sieve. The schematic illustration of the montmorillonite activation process is presented in Figure 2.



Figure 2. Illustration of MMT activation

Characterization

The activated and pre-activated zeolite was then analyzed for its metal oxide content using Thermo Fisher Scientific's XRF with x-ray path: air, Eff. Stationer: 13.0 mm and Eff. Area : 132.7 mm², sample height : 3.77 mm with a mass of 500 mg. Furthermore, the crystalline phase of zeolite was evaluated using a powder X-Ray diffractometer (XRD, Shimadzu 7000) with CuK α radiation (λ = 1.5405 Å) operated at a scanning range of 20-80 configuration. The crystallite domain size was calculated using the Debye-Scherrer formula as shown in Equation (1).

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \quad (1)$$

C. Results and discussion

Based on the XRF analysis presented in Table 1, the natural MMT obtained from Gowa Regency contains several metal oxides, with Fe_2O_3 identified as the dominant impurity compound. The Fe_2O_3 content in natural MMT reached 31.61%, which was considerably higher than other oxide components such as Al_2O_3 , TiO_2 , and CaO . This result indicates that the natural montmorillonite contains a significant amount of iron-bearing minerals that are commonly associated with clay deposits formed through weathering and sedimentation processes. The high Fe_2O_3 content may influence the physicochemical properties of montmorillonite, including color, adsorption capacity, and catalytic performance.

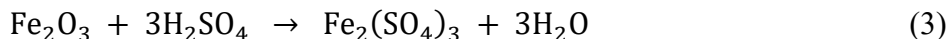
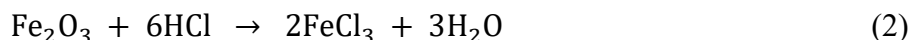
Table 1. Results of analysis of metal oxide content in MMT before and after activation

Metal Oxide	Natural MMT	MMT purified HCl	MMT purified H_2SO_4
Fe_2O_3	31.61	21.78	18.83
SiO_2	39.51	40.13	41.02
Al_2O_3	9.97	15.87	19.82
TiO_2	7.51	8.89	7.14
ZnO	1.25	1.36	1.36
K_2O	3.67	3.47	3.24
MgO	1.22	1.09	1.09
CaO	1.98	1.01	1.14
Cr_2O_3	0.72	2.23	2.32
ZrO_2	0.93	1.84	1.92
CuO	0.43	0.49	0.53
Nb_2O_5	0.45	0.48	0.39
CuO	0.43	0.91	0.88
SrO	0.32	0.45	0.32

After acid activation using HCl and H_2SO_4 , the Fe_2O_3 content decreased significantly to 21.78% and 18.83%, respectively. The reduction in Fe_2O_3 concentration demonstrates that inorganic acid activation effectively dissolved iron oxide impurities from the montmorillonite structure. Among the acids used, H_2SO_4 exhibited a greater ability to remove Fe_2O_3 compared to HCl, indicating a stronger dealumination and demineralization effect during the activation process (Huang et al., 2026). This phenomenon occurs because acid treatment promotes the leaching of exchangeable cations and impurity oxides from the interlayer and surface structure of montmorillonite.

The decrease in Fe_2O_3 content after acid activation suggests that inorganic acids promoted the dissolution of iron oxide impurities from the montmorillonite structure. During the activation process, hydrogen ions (H^+) originating from HCl and H_2SO_4 penetrated the interlayer structure of montmorillonite and replaced exchangeable cations such as Ca^{2+} , Mg^{2+} , K^+ , and Fe^{3+} (Side et al., 2023). This ion-exchange process weakened the bonding between metal oxides and the aluminosilicate framework, resulting in partial dissolution of impurity oxides. The proposed mechanism begins with protonation of oxygen atoms in the clay structure, which increases the susceptibility of metal–oxygen bonds toward hydrolysis. Iron oxide impurities are subsequently dissolved through acid-mediated reactions, as illustrated in Equations (2) and (3). These reactions explain the significant reduction in Fe_2O_3 content after activation, especially in the sample treated with H_2SO_4 . Sulfuric acid showed stronger dissolution capability because sulfate ions may enhance the destabilization of iron-containing minerals within the montmorillonite matrix. In addition to iron

oxide dissolution, acid activation also promoted partial removal of octahedral aluminum ions (dealumination), leading to relative enrichment of silica content in the activated montmorillonite.



In addition to the decrease in Fe_2O_3 content, the SiO_2 concentration slightly increased after acid activation, from 39.51% in natural MMT to 40.13% and 41.02% after HCl and H_2SO_4 treatments, respectively. This increase suggests that acid activation selectively removed impurity oxides while enriching the silica framework of montmorillonite. Similar trends were also observed for Al_2O_3 , where the oxide content increased after activation, indicating structural modification and purification of the clay mineral (Putri et al., 2023a, 2023b). The present findings are consistent with previous studies reporting that Fe_2O_3 is commonly the major impurity found in natural clay minerals and can be significantly reduced through acid activation (Rahman et al., 2023). Furthermore, several studies have reported that sulfuric acid tends to provide stronger dissolution of iron-containing minerals compared to hydrochloric acid because sulfate ions enhance the breakdown of metal oxide bonds within the clay structure (H. Zhou et al., 2025).

The XRD analysis results obtained in this study are consistent with several previous studies related to acid-activated montmorillonite and natural clay minerals. The diffraction peak at $2\theta \approx 26.6^\circ$ corresponding to quartz silica (SiO_2 , JCPDS No. 46-1045). It is similar with previous study, reported that that acid activation increased the relative intensity of silica peaks due to the dissolution of impurity oxides and structural purification of montmorillonite (Bejar et al., 2016). This finding supports the present XRF results, where the SiO_2 content increased after HCl and H_2SO_4 activation. Furthermore, this finding indicates the acid treatment caused significant modification of the montmorillonite microstructure, leading to improved adsorption properties and reduced iron oxide impurities. Similar behavior was observed in the present research, where the Fe_2O_3 diffraction peaks at $2\theta \approx 35.6^\circ$ and 54.1° (hematite phase, JCPDS No. 33-0664) decreased substantially after acid activation. This reduction corresponds well with the XRF data showing a decrease in Fe_2O_3 content from 31.61% in natural MMT to 18.83% after H_2SO_4 activation.

Furthermore, research conducted by (Side & Putri, 2022) on natural clay from different Pattallassang sub-district Gowa Regency reported that the dominant crystalline phases consisted mainly of silica and aluminosilicate minerals with considerable iron oxide impurities. Compared with the previous work, the present study provides more comprehensive evidence showing that inorganic acid activation successfully reduced hematite crystallinity while enhancing silica and alumina dominance in the montmorillonite structure.

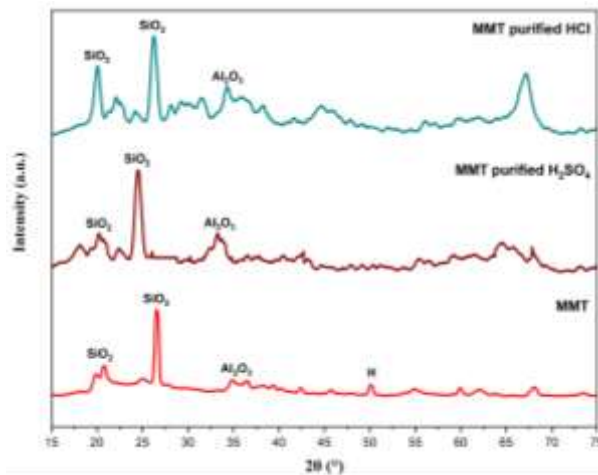


Figure 3. The diffractogram of natural MMT and purified MMT

In addition, the decrease in crystallite size after acid activation observed in this study is in agreement with the findings reported by previous study which reported that acid leaching promotes structural fragmentation and partial exfoliation of clay minerals ([Ria et al., 2025](#)). The reduction in crystal size occurs because acid attack weakens the metal–oxygen bonds within the aluminosilicate framework, resulting in smaller crystallite domains and increased surface area. Such structural modification is beneficial for adsorption and catalytic applications because smaller crystallite sizes provide more active surface sites.

Table 2. The average crystallite size of natural MMT and purified MMT

Sample	Phase	2θ (°)	FWHM (β)	Crystal plane (hkl)	Average crystallite size (nm)
MMT	SiO ₂ (Quartz)	20.1	0.82	(100)	41.2
	SiO ₂ (Quartz)	26.6	0.38	(101)	
	Al ₂ O ₃	34.2	0.91	(104)	
	Hematite (Fe ₂ O ₃)	50.1	0.76	(024)	
MMT purified HCl	SiO ₂ (Quartz)	20.0	0.95	(100)	35.1
	SiO ₂ (Quartz)	26.5	0.44	(101)	
	Al ₂ O ₃	34.0	1.02	(104)	
MMT purified H ₂ SO ₄	SiO ₂ (Quartz)	20.2	1.01	(100)	29.9
	SiO ₂ (Quartz)	24.4	0.41	(101)	
	SiO ₂ (Quartz)	24.4	0.41	(101)	
	Al ₂ O ₃	34.1	1.10	(104)	

Another relevant study reported that natural clay minerals from South Sulawesi contained high iron-bearing phases that could influence the adsorption performance and crystallinity of the material (Rahman et al., 2023). Their findings support the current observation that hematite is one of the major impurity phases in natural montmorillonite and can be effectively reduced through acid activation. Overall, the present study demonstrates similar trends to previous investigations, particularly regarding the reduction of hematite impurities, enhancement of silica crystallinity, and structural modification after acid treatment. However, this work provides additional insight into the comparative effectiveness of HCl and H₂SO₄ activation on natural montmorillonite from Gowa Regency, especially in relation to changes in oxide composition, crystalline phase intensity, and crystallite size.

D. Conclusion

In this study, natural montmorillonite from Gowa Regency was successfully activated using inorganic acids, namely HCl and H₂SO₄, to modify its metal oxide composition and crystalline structure. XRF analysis demonstrated that Fe₂O₃ was the dominant impurity oxide in natural montmorillonite, and its concentration decreased significantly after acid activation, particularly in the H₂SO₄-treated sample. Meanwhile, the relative content of SiO₂ and Al₂O₃ increased after activation, indicating effective purification and structural modification of the montmorillonite framework. XRD analysis confirmed the presence of quartz silica, alumina, and hematite phases, where the intensity of hematite peaks decreased after acid treatment due to dissolution of iron oxide impurities. Furthermore, the average crystallite size decreased from 41.2 nm in natural montmorillonite to 35.1 nm and 29.9 nm after HCl and H₂SO₄ activation, respectively, suggesting structural fragmentation and increased amorphous characteristics. Among the acids used, H₂SO₄ exhibited greater effectiveness in removing impurity oxides and reducing crystallite size compared to HCl. Overall, inorganic acid activation significantly improved the physicochemical characteristics of natural montmorillonite from Gowa Regency, making it a promising material for future adsorption, catalytic, and environmental remediation applications. This study also provides important scientific insight into the effect of inorganic acid treatment on the structural transformation of local Indonesian clay minerals.

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