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1	Delamination of kaolinite-potassium acetate intercalates by ball-milling
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3 1	Hongfei Cheng ^{a,b,c} Oinfu Liu ^a Jinshon Zhong ^b
4 5	Jing Vang ^c and Ray L. Frost ^{co}
6	ong rang and ray 2. 1105t
7	^a School of Geoscience and Surveying Engineering, China University of Mining &
8	Technology, Beijing 100083, China
9	
10	^b School of Mining Engineering, Inner Mongolia University of Science & Technology,
11	Baotou 014010, China
12	
13	^c Chemistry Discipline, Faculty of Science and Technology, Queensland University of
14	Technology, 2 George Street, GPO Box 2434, Brisbane, Queensland 4001, Australia
15	
16	
17	Corresponding Author:
18	Derr I. Enert
19 20	Ray L. FTUST P +61 7 3138 2407
21	M: +61 7 414 84 2407
22	F: +61 7 3138 1804
23	E: r.frost@qut.edu.au

[•] Author for correspondence (<u>r.frost@qut.edu.au</u>) P: +61 7 3138 2407 F: +61 7 3138 1804

24 25 26 27 28 29	Delamination of kaolinite–potassium acetate intercalates by ball-milling Hongfei Cheng ^{a,b,c} , Qinfu Liu ^a , Jinshan Zhang ^b , Jing Yang ^c and Ray L. Frost ^{c•}
30	^a School of Geoscience and Surveying Engineering, China University of Mining & Technology, Beijing
31	100083, China
32	
33	^b School of Mining Engineering, Inner Mongolia University of Science & Technology, Baotou 014010,
34	China
35	
36	^c Chemistry Discipline, Faculty of Science and Technology, Queensland University of Technology, 2
37	George Street, GPO Box 2434, Brisbane, Queensland 4001, Australia
38	
39	Abstract:
40	Structural changes in intercalated kaolinite after wet ball-milling were examined by
41	scanning electron microscopy (SEM), X-ray diffraction (XRD), specific surface area
42	(SSA) and Fourier Transform Infrared spectroscopy (FTIR). The X-ray diffraction pattern
43	at room temperature indicated that the intercalation of potassium acetate into kaolinite
44	causes an increase of the basal spacing from 0.718 to 1.42 nm, and with the particle size
45	reduction, the surface area increased sharply with the intercalation and delamination by
46	ball-milling. The wet ball-milling kaolinite after intercalation did not change the
47	structural order, and the particulates have high aspect ratio according SEM images.
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[•] Author for correspondence (<u>r.frost@qut.edu.au</u>) P: +61 7 3138 2407 F: +61 7 3138 1804

49 Keywords: Kaolinite; Potassium acetate; Intercalation; Delamination; Particle size

50 1. Introduction

Kaolinite has a wide variety of applications in industry, particularly as paper filler 51 52 and coating pigment [1-6]. It is used as an extender in aqueous based paints and inks, a functional additive in polymers and is the major component in ceramics [7-9]. Kaolinite 53 is an inexpensive additive which is able to form stable dispersions and to improve the 54 properties of the material. More recently, this mineral has been found with increasing 55 usage in other applications such as a petroleum cracking catalyst, and a filler in adhesives 56 and plastics [10]. Properties of kaolinite, particularly important for industrial applications, 57 58 are particle size distribution, particle shape, structural order-disorder and crystallinity, specific surface area and whiteness [5]. These properties can be enhanced with several 59 treatments such as dry-grinding, intercalation and delamination/exfoliation [9, 11, 12]. 60

61

62 Lately, some methods for intercalation of kaolinite, in which the interlayer space the layered kaolinite particles were intercalated with small molecules such as urea, potassium 63 acetate, dimethylsulphoxide and so on [13-16]. Meanwhile, delamination of kaolinite is 64 an important industrial procedure, which influences the rheological properties of 65 dispersions used in the ceramic industry, the coating properties of the kaolinite used in the 66 paper industry and gas barrier properties of rubber [17]. The delamination/exfoliation can 67 increase the usability of kaolinite reserves by decreasing the particle size and therefore 68 increasing the specific surface area (SSA) of kaolinite. 69

70

In the past few years, great interest has been expressed in research to increase the specific surface area [3, 17-21]. This surface reactivity can be enhanced through particle

size reduction, which traditionally can be achieved by grinding (either wet or dry) [3, 22]. 73 74 Often mechanically ground clay minerals are frequently used in industry. Moreover, an innovative technique for decreasing the particle size of clay minerals has been proposed. 75 The previous researches reported the structural degradation of kaolinite is observed, the 76 morphology of kaolinite is damaged and the ratio of the diameter to thickness is very low 77 by the mechanochemical treatment [23]. It is reported that delamination after 78 79 intercalation was an effective method for reducing the particle-size of kaolinite to the 80 micronic range, while the crystalline structure and lamellar morphology are still retained [3, 11]. In this work, the effect of intercalation and ball-milling on kaolinite is extensively 81 82 studied. The Scanning electron microscopy (SEM), X-ray diffraction (XRD), Specific surface area and infrared spectroscopy (IR) are used to investigate the changes in the 83 particle-size, crystallinity, and morphology of kaolinite. 84

85

86 2. Experimental methods

87 2.1 Materials

The sample used in this study was the natural kaolinite from Zhangjiakou, Hebei province of China, with particle size of 45 μ m. Its chemical composition in wt% is SiO₂ 44.64, Al₂O₃ 38.05, Fe₂O₃ 0.22, MgO 0.06, CaO 0.11, Na₂O 0.27, K₂O 0.08, TiO₂ 1.13, P₂O₅ 0.13, MnO 0.002, loss on ignition 15.06. The major mineral component is well ordered kaolinite (95 wt %) with a Hinckley index of 1.31. The potassium acetate (A.R.) was obtained from the Beijing Chemical Reagents Company, China.

94

95 **2.2 Samples preparation**

96 The potassium acetate (KAc) intercalated compound was prepared by adding 1.05

97 kg of kaolinite into 2.45 Kg of KAc solution at a mass percentage concentration of 30 %, 98 stirring for 10 min. After aging for 24 h, a GF-1100 type machine, filled with zircon balls (zirconium dioxide ca. 60 % and silicon dioxide ca. 40% by mass) for multi-purpose 99 high-speed dispersion, was used to grind the KAc intercalated kaolinite slurry for 2h at 100 room temperature. This machine was purchased from Jiangyin Shuangye Machinery 101 Equipment CO. Ltd. The diameter of zircon balls is from 0.8 to 1.2 mm. The zircon balls 102 103 and the ground slurry were separated by a mesh, and the delaminated kaolinite was 104 recovered by filtration from the ground slurry. A comparative study was taken on the delamination kaolinite without intercalation. Kaolinite slurry without intercalation was 105 106 obtained from kaolinite and water with a ratio of 1:3. The slurry was aged 24 h, and then ground by zircon balls and recovered by the same process mention above. 107

108 The samples were allowed to dry at room temperature before the SEM, XRD and109 FT-IR analysis.

110

111 **2.3 Characterization**

The morphology of kaolinite particles was observed by using a scanning electron 112 113 microscope (SEM), Hitachi S-4800. Samples were coated with a gold/palladium film and the SEM-images were obtained using a secondary electron detector. The powder X-ray 114 diffraction (XRD) analysis was performed using a Japan Rigaku D/max-rA X-ray 115 diffractometer (40 kV, 100 mA) with Cu (λ =1.54178 Å) irradiation at the scanning rate of 116 117 2 °/min in the 2θ range of 2.6-50 °. The Specific Surface Area (SSA) values were obtained with an automatic system (Model No. 2200 A, Micromeritics Instrument Corp., 118 119 Norcross, GA) at liquid-nitrogen temperature, using the BET method. Nitrogen was used 120 as the adsorbate. Before measurement, the samples were pre-heated at 80 °C under 121 nitrogen for ca. 24 h. The specific surface area was calculated by the BET equation and the total pore volumes were evaluated from nitrogen uptake at relative pressure of ca. 122 0.99. The particle size distribution was determined by low angle laser light scattering 123 (Lalls, Mastersizer Model, Malvern). The measurements were performed at 25 °C on 124 dispersions of about 10 mg of samples in 50 ml of distilled water. The delamination 125 kaolinite after intercalation and the kaolinite only by ball milling were dispersed using 126 127 ultrasound bath. Fourier transform infrared (FT-IR) spectroscopic analysis was undertaken using a NICOLET 750 SX spectrometer. FT-IR spectra between 500 and 4000 128 cm⁻¹ were obtained. The samples were prepared at KBr pellets (ca. 2 % by mass in KBr). 129

130

131 **3. Results and discussion**

132 **3.1 X-ray diffraction**





134 Figure 1 the XRD patterns of (a) original kaolinite, (b) kaolinite intercalated by KAc, (c) kaolinite

135

136

ball-milling after intercalation

Fig. 1 shows the XRD patters of original kaolinite, kaolinite intercalation compound 137 with KAc and the delamination kaolinite. The XRD pattern of kaolinite intercalation 138 compound shows a large difference from the original untreated kaolinite because of the 139 140 KAc intercalation. The effect of intercalation causes a decrease in the position of the (001) reflection. The effect of ball-milling causes the loss of the intensity at 1.42 nm and after 2 141 h of milling, no intensity remains in this peak. The intensity decrease of the (001) 142 143 reflection shows that the kaolinite layers are exfoliated and delaminated. The intercalation 144 caused the destruction of the hydrogen bonding between the kaolinite layers [24].

A previous study reported the method of intercalation and delamination/exfoliation 145 146 treatment of kaolinite induces the structure degradation of kaolinite [25]. The degree of structural order/disorder of the kaolinite samples can be estimated by XRD. It is reported 147 that an increase of the structural disorder caused an obvious weakening of reflections 111 148 and 021 (2 θ between 17 and 27°), which were replaced by a broad peak of scattering with 149 150 weak modulations [14, 26, 27]. However, in this study, no broad peak was found in the pattern of kaolinite intercalation compound; instead, the reflections 022, $1\overline{3}0$, $\overline{1}31$, 003, 151 $1\overline{3}1$ and $\overline{1}13$ can be found in the pattern of the delaminated kaolinite, which suggests 152 153 that the well crystallized kaolinite almost did not undergo structural degradation after intercalation and ball-milling. The Hinckley index of the samples is shown in Table 1. 154

155

156 **3.2 Scanning electron microscopy (SEM)**

Fig. 2 displays the SEM images of the original kaolinite, the intercalated kaolinite and the ball milling samples without or with intercalation. The morphology of kaolinite (Fig.2a) indicates book-like structures. The distance of adjacent layers is expanded after intercalated by KAc (Fig.2b). The reduction of particle size of kaolinite by ball milling after intercalation is evident in the SEM imagines (Fig. 2d). It is possible to verify in Fig. 2d that the book-like structured kaolinite has been delaminated and the individual particles are randomly distributed without forming apparent aggregation. The particles of the ball milled kaolinite with intercalation are less than 2 μ m, forming a layered morphology with individual platelets.

166





168 **Figure 2** The SEM images of (a) original kaolinite, (b) kaolinite intercalated by potassium acetate, (c)

169 kaolinite ball-milling without intercalation and (d) kaolin by intercalation and ball-milling

170

Previous studies suggest that the morphology of kaolinite is thick stacks and the sheet-type structure of the kaolinite is retained after mechanochemical treatment [28, 29]. A comparison of Fig.2c and d gives new discovery that not only the particle size of kaolinite after intercalation and ball milling is smaller than the one treated only by ball milling, but also less agglomerates and higher lamellarity than treatment only by ball milling. In the rubber industry, kaolinite with high lamellarity is principally used as reinforcing fillers and processing aids. It reduces the diffusion rate of gases and liquids in vulcanised rubber. This is due to their platy morphology, which increases the diffusion path [30].

As a consequence of this delamination/exfoliation, the platelets look less elongated and show the typical euhedral, hexagonal morphology. Although the form of the individual particles has been strongly modified, the layered morphology is preserved and the particle size reduction is very clearly after intercalation and ball milling. The platelets of kaolinite after intercalation and delamination have high aspect.

185

186 **3.3 Specific surface area measurements**

The reduction in the particle size is clearly evident in the SEM (Fig.2) and Table 1. The particle distribution of the original kaolinite is about 45 μ m. The decrease of particle size obtained under the experimental condition of treatment that kaolinite without intercalation ball milled is lower. The particle size of kaolinte by intercalation decreased to less than 2 μ m with 39 %. After ball milling, the kaolinite by intercalation were delaminated, and the particle size of the lamellar was reduced to less than 2 μ m with 98%.

Table 1 includes the particle size (PS) and specific surface area (SSA) for original kaolinite, intercalation kaolinite, ball-milled kaolinite without intercalation and ball-milled kaolinite after intercalation. The original kaolinite has a value that is 8.78 m^2/g . After intercalation the SSA is 12.57 m^2/g . The SSA increase to 27.52 m^2/g after intercalation and ball-milled. The value of kaolinite SSA after ball-milled without interclation increase is less than that intercalation and ball-milling.

200

201 **3.4 FTIR analysis**

The FTIR spectra of kaolinite and its exfoliations show four important 202 OH-stretching bands at (v_1) 3695, (v_2) 3668, (v_3) 3650 and (v_5) 3620 cm⁻¹, which can be 203 observed in Fig.3. The band (v_1) at 3695 cm⁻¹ is assigned to the in-phase hydroxyl 204 stretching vibration of the inner surface hydroxyl; the bands at 3650 and 3668 cm⁻¹ are 205 assigned to the out-of--phase hydroxyl stretching vibration of the inner surface hydroxyls 206 The outer hydroxyl units are situated on the surface of the lamellae, which are accessible 207 for hydrogen bonding with the appropriate intercalating molecules. The band at (v_5) 3620 208 cm⁻¹ is attributed to the stretching frequency of the internal (inner) hydroxyl groups of 209 kaolinite, which lie within the lamellae in the plane common to both the tetrahedral and 210 211 octahedral sheets. Being within the layers, the inner hydroxyl cannot participate in hydrogen-bonding to adsorbed molecules. So, the inner hydroxyl stretching band is not 212 usually influenced by the interlayer modification of kaolinite [31-33]. 213

214

The results of the band component analysis of the infrared spectra are reported in Table 2. The decrease in the intensity of both (v_2) 3668 cm⁻¹ and (v_3) 3650 cm⁻¹ bands is illustrated in Fig.3. There is an apparent exponential decrease in the intensity of the hydroxyl stretching bands for the ball milled kaolinite without intercalation, whereas the intensity almost has no change after intercalation and delamination. This can be related to the loss of some OH groups though a mechanical grinding, which occurred in the newly external surface generated in the particle size reduction process [5]. The decreasing intensity and the broadening of the OH stretching bands indicate structural deterioration caused by grinding without intercalation.



Figure 3 The FTIR spectra in 3750-3550 cm⁻¹ region Fig. 4 the FTIR spectra in 600-1200 cm⁻¹ region of (a) of (a) kaolinite, (b) kaolinite ball-milling without kaolinite, (b) kaolinite ball-milling without intercalation intercalation and (c) kaolinite ball-milling after and (c) kaolinite ball-milling after intercalation intercalation

224

According to Farmer[34] and Franco[3], delamination can be examined from the intensity and position of the Si-O vibrational bands which gives a dipole oscillation perpendicular to the plates. Fig. 4 shows in the 1200-600 cm⁻¹ region, the FTIR spectra of original kaolinite, kaolinite ball milling without intercalation and kaolinite delamination after intercalation. New and low intensity bands appear at low wavenumbers. The bands at (v_{10}) 1115 cm⁻¹ and (v_{15}) 940 cm⁻¹ are attributed to the hydroxyl deformation of the inner surface and the Si-O out of plane vibrations. The lack of these two bands and decreasing intensity of bands (v_{11}) 1099 cm⁻¹ and (v_{16}) 926 cm⁻¹ are also an indication that the damage to structure of kaolinite happened after ball milling without intercalation. After intercalation with KAc solution, these changes are not observed. This is also an indication that there was less damage to the structure of kaolinite in delamination after intercalation, consistent with the results of XRD and SEM.

237

238 4. Conclusions

Delamination of kaolinite was achieved through intercalation and ball-milling. The 239 changes in the molecular structure of kaolinite were confirmed by XRD, SEM and FT-IR. 240 The proportion of the particles ($< 2 \mu m$) increases sharply by intercalation and ball milling, 241 and at the same time the proportion of particles ($<1\mu m$) is progressively increased. As a 242 consequence of this particle-size reduction, the SSA increases from 8 to 27 m^2/g after 243 244 intercalation and ball milling. Moreover, no evidence of change of the structural order of kaolinite has been detected from the XRD and FTIR studies. The platelets of kaolinite 245 after intercalation and delamination have higher diameter-thickness ratio and ideal 246 layered morphology. 247

248

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293	Table 1 Hincklev	v index. n	oarticle size an	d specific surface	area data of	'original kaolin.
				······································		,

294 intercalation kaolin, exfoliation kaolin after intercalation and without intercalation.

- 296 **Table 2 Band component analysis kaolin, exfoliated kaolin without intercalation and**
- 297 exfoliated kaolin intercalated by KAc

298Table 1 Hinckley index, particle size and specific surface area data of original

299 kaolinite, intercalation kaolinite, exfoliation kaolinite after intercalation and without

300 intercalation.

Kaalinita samplas	Hinckley	PS	PS	SSA(m ² /
Kaolinite samples	Index	(<1µm)	(<2µm)	g)
Original kaolinite	1.305	5%	18%	8.78
Intercalation kaolinite	1.297	12%	39%	12.57
Ball milling kaolinite without intercalation	0.852	15%	57%	13.54
Ball milling kaolinite after intercalation	1.225	75%	98%	27.52

302 Table 2 Band component analysis kaolinite, exfoliated kaolinite without

Band	Original	al kaolinite		Exfoliat without	Exfoliated kaolinite without intercalation			Exfoliated kaolinite intercalated by KAc		
parameters	Center	FWHM	%	Center	FWHM	%	Center	FWHM	%	
v_1	3694	21.8	6.79	3695	20.0	6.58	3695	23.0	7.18	
v_2	3668	15.8	1.18	3669	15.8	0.50	3668	19.5	1.25	
V3	3651	20.6	2.29	3652	27.6	1.38	3651	23.6	2.14	
v ₅	3620	8.8	2.36	3620	9.1	2.16	3620	13.1	1.89	
v_{10}	1115	12.0	1.90				1113	24.0	6.59	
v_{11}	1099	45.8	13.86							
V ₁₂	1045	38.8	11.11	1083	55.1	10.48	1093	48.3	16.23	
V ₁₃	1032	18.8	13.97	1033	29.0	17.12	1033	27.6	19.93	
v_{14}	1009	20.0	12.21	1008	20.3	13.87	1008	18.7	9.21	
v_{15}	940	17.6	3.54				939	19.5	2.26	
v_{16}	926	12.2	1.01				925	11.8	1.00	
v_{17}	913	18.9	5.61	914	19.5	6.27	914	21.9	6.67	
v_{18}	791	21.3	0.72	792	26.8	1.10	792	26.5	1.18	
V19	756	23.1	0.81	756	22.2	0.80	756	23.2	0.91	
v_{20}	695	29.2	2.55	697	26.6	2.52	696	28.3	2.88	
V21				590	35.4	1.40	591	37.3	1.50	

303	intercalation and exfoliated kaolinite intercalated by KAc
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kaolin exfoliated after intercalation 308

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Fig 1 the XRD patterns of (a) original kaolinite, (b) kaolinite intercalated by KAc, (c)

319 kaolinite delamination after intercalation

320





- 323 potassium acetate, (c) kaolinite by ball milling without intercalation and (d)
- 324 kaolinite by intercalation and delamination





³²⁶ Fig. 3 the FTIR spectra in 3750-3550 cm⁻¹ region of (a) kaolinite, (b) kaolinite ball

327 milling without intercalation and (c) kaolinite delamination after intercalation





329 Fig. 4 the FTIR spectra in 600-1200 cm⁻¹ region of (a) kaolinite, (b) kaolinite ball

