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Micro-Structure Differences in Kaolinite Suspensions

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Micro-Structure Differences in the Kaolinite Suspensions

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Abstract

SEM observations of the aqueous suspensions of kaolinite from Birdwood (South Australia) and Georgia (USA) show noticeable differences in number of physical behaviour which has been explained by different microstructure constitution.. Birdwood kaolinite dispersion gels are observed at very low solid loadings in comparison with Georgia KGa-1 kaolinite dispersions which remain fluid at higher solids loading. To explain this behaviour, the specific particle interactions of Birdwood kaolinite, different from interaction in Georgia kaolinite have been proposed. These interactions may be brought about by the presence of nano-bubbles on clay crystal edges and may force clay particles to aggregate by bubble coalescence. This explains the predominance of stair step edge-edge like (EE) contacts in suspension of Birdwood kaolinite. Such EE linked particles build long strings that form a spacious cell structure. Hydrocarbon contamination of colloidal kaolinite particles and low aspect ratio are discussed as possible explanations of this unusual behaviour of Birdwood kaolinite. In Georgia KGa-1 kaolinite dispersions instead of EE contact between platelets displayed in Birdwood kaolinite, most particles have edge to face (EF) contacts building a cardhouse structure. Such an arrangement is much less voluminous in comparison with the Birdwood kaolinite cellular honeycomb structure observed previously in smectite aqueous suspensions. Such structural characteristics of KGa-1 kaolinite particles enable higher solid volume fractions pulps to form before significantly networked gel consistency is attained.

Key words: clay aggregates, kaolinite microstructure, Georgia Kaolinite, Birdwood Kaolinite, colloids, clay gelation

Introduction

All contemporary approaches for controlling clay suspension behaviour are based on the theory of colloid stability developed by Derjaguin and Landau [1], and Verwey and Overbeek [2]. This theory, known as DLVO theory, is where competing repulsive electrostatic and attractive van der Waals forces determine if a particular colloid clay suspension will be stable (in sol form) or coagulate (in gel form). On the basis of DLVO theory Hogg [3] described opportunities for the control of floc characteristics by physical displacement between particles, chemical changes in pore electrolyte, and introduction of a new mineral phase into the system. All these manipulations may collapse the electrical double layer, which will lower the electrokinetic potential that may bring particles close enough to allow the Van der Waals forces to bond particles into larger aggregates and significantly speed up the sedimentation rate.

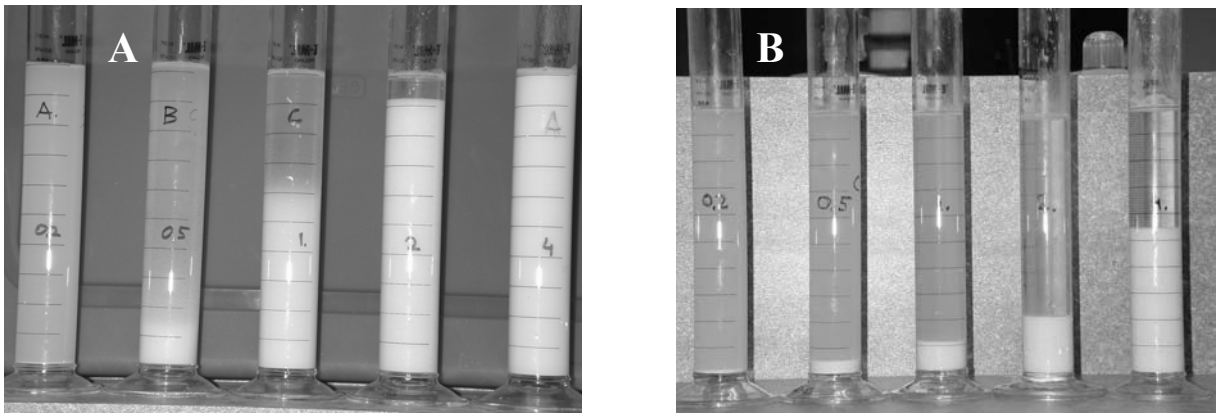

Fig. 1 Bench test of different concentration of solids in (A) Birdwood kaolinite and (B) Georgia kaolinite suspensions in the pH 8.

However not all kaolinite suspensions produce sols which display DLVO behaviour, and some of them, like kaolinite from Birdwood in South Australia [4], show unusual behaviour and gel at very low solid loadings. Similar behaviour was reported in suspensions of natural oil-containing kaolinite from Canada [5] and Australian kaolinite associated with coal deposits [6] which was recognised as the major cause of difficulties in flocculating these suspensions.

An example how differently kaolinites may behave in aqueous suspension is shown in the bench test Fig. 1. This tests presented shows differences in the settling behaviour between Birdwood (A)

and Georgia KGa-1 (B) kaolinite suspensions which differ in solids loading (from left to the right, 0.2 wt%, 0.5 wt%, 1 wt%, 2 wt%, and 4 wt%). At a very low loading content (0.2 wt%) particles in both kaolinites are well disperse and only larger crystals settled to the bottom of the cylinder. When solid content is increased (0.5 wt% and 1 wt%) a gelling mass forms at the bottom of the cylinders with some particles remaining dispersed in the suspension above the gelling mass. Gelling is more advanced in the case of Birdwood kaolinite (Fig. 1A) When solids concentration exceeds a certain value the suspension gels and locks large and minute particles in a three-dimensional network leaving clear supernatant above the compacting gel (2 wt%). Inside the gelled part of suspension free settling is not observed but more or less restricted hindered compaction takes place. Denser suspensions (4 wt%) are very slowly to compact and in case of the Birdwood kaolinite the gelled sample resists to settle.

In the references [5, 6] it has been suggest that particles in such gels are in constant contact with each other by creating a three-dimensional structure. Such a structure was also reported in the first ever SEM observations of the clay particle in dilute suspensions [6] where kaolinite platelets were found to form contacts with surrounding particles in a three-dimensional network. In this work, dilute suspensions of two different kaolinites microstructural properties were investigated with focus on particle mutual arrangements. These mutual particles arrangements create aggregate 3-D microstructure which is responsible for suspension macroscopical behaviour. Present investigation will test this hypothetical statement.

Experimental

The material used for this study was commercially available K15GM kaolinite from Birdwood in South Australia [4] and Georgia kaolinite, KGa-1. The latter was obtained from the Clay Minerals Society; and is a fully characterised sample [7].

Dry kaolinite was mixed with aqueous 10^{-2} M NaCl electrolyte at high solid content by shaking in a SPEX mill for 5 min. The slurries were then diluted to the required solid concentrations (4 wt.%) and stirred for 2 h using a magnetic stirrer.

For the microstructural studies of the suspensions, cryo-transfer method of sample preparation was used [8] to avoid structural rearrangement caused by surface tension during oven or room

temperature drying. The vitrified samples were kept in a frozen state and placed onto the liquid nitrogen cooled specimen stage of the Philips XL30 Field Emission Gun Scanning Electron Microscope with Oxford CT 1500HF Cryo-stage. The samples were fractured under vacuum and sublimated after which they were coated with gold and palladium to a thickness of 3 nm. The samples were then examined in the SEM using a cathode voltage of 5 kV.

A transmission X-ray microscope with 60 nm tomographic resolution has been installed at beamline BL01B[2] of NSRRC in Taiwan. This device has a superconducting wavelength shifter source, which provides a photon flux of 5 x 1012 photons/s/0.1 % bw in the energy range 5-20 keV. X-rays generated by a wavelength shifter are primarily focused at the charge coupled detector by a toroidal focusing mirror with focal ratio nearly 1:1. A double crystal monochromator exploiting a pair of Ge (111) crystals selects X-rays of energy 8-11 keV. After passing through the focusing mirror and double crystal monochromator, the X-rays are further shaped by a capillary condenser. Its entrance aperture is about 300 μm, with an end opening about 200 μm and is 15 cm long. This capillary condenser gives a reflection angle of 0.5 mrad with respect to the propagation direction. The condenser intercepts the impinging X-rays and further focuses them onto the sample with a focusing efficiency as high as 90% due to the totally reflecting nature inside the capillary. The zone-plate is a circular diffraction grating consisting of alternating opaque and transparent concentric zones. In the microscope, the zone-plate is being used as an objective lens magnifying the images 44 × and 132× for the first order and third order diffraction mode, respectively. Conjugated with a 20× downstream optical magnification, the microscope provides total magnification of 880× and 2640× for first order and third diffraction order mode, respectively. The phase term can be retrieved using the Zernike's phase contrast method that was introduced in light microscopy since the 1930s. The gold-made phase ring positioned at the back focal plane of the objective zone-plate retards or advances the phase of the zeroth- order diffraction by $\pi/2$, resulting in a recording of the phase contrast images at the detector.

Electrokinetic potential in both samples was measured in function of pH using AcoustoSizer II (colloidal Dynamics) in 0.01 M NaCl in 6 wt % kaolinite suspensions and particle size distribution was determined from Mastersizer (Malvern Instrument) apparatus.

Results and discussions

The grain size investigation show that both kaolinites are similar in dispersion, however the specific surface area found by N_2 –BET was 19.7 m^2/g in case of Birdwood kaolinite [9] and 15.3 m^2/g in case of Georgia kaolinite [7] suggesting that the Birdwood kaolinite contained slightly finer particles.

Differences in the sedimentation behaviour between the kaolinite samples were displayed and were most probably caused by differences in particle electrostatic charge and morphology as well as the texture pattern when networking in a three-dimensional structure. Consequently the morphology of kaolinite samples was studied using SEM (Fig. 2).

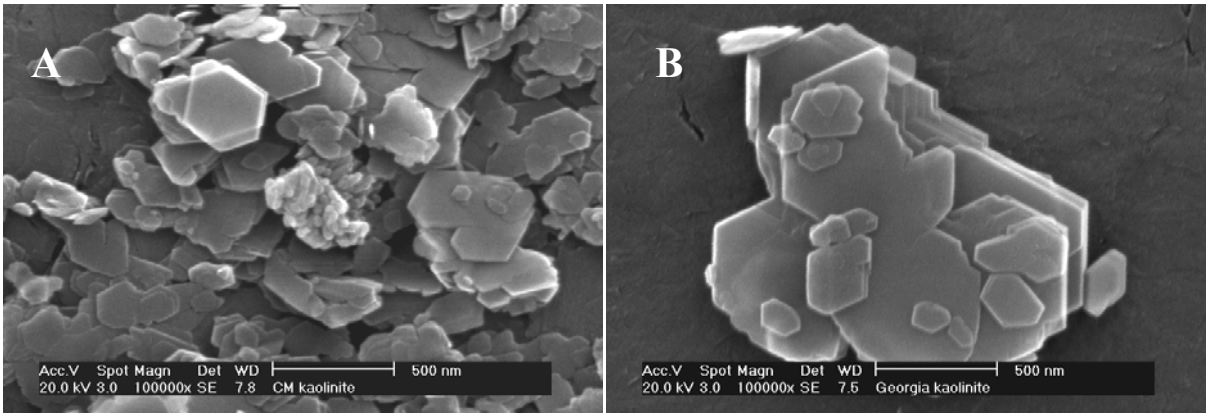

Fig. 2 High resolution SEM images of (A)- Birdwood kaolinite finest colloidal fraction, (B)- Georgia KGa-1 kaolinite finest colloidal fraction.

The colloidal fraction of Birdwood kaolinite consists of pseudo hexagonal euhedral crystals with dimensions down to ~50 nm (Fig. 2A). The crystals typically are thin and flexible plates of very high aspect ratio (ratio of average diameter to platelet thickness is above 12). SEM investigations of fine (below 2 μm) fraction of Birdwood kaolinite show that crystals are on average 160 nm in diameter (150 particles measured) and about 13 nm thick. SEM micrographs of the Georgia KGa-1 kaolinite (Fig. 2B) also show pseudo hexagonal euhedral crystals but the platelets in KGa-1 kaolinite appear to be thicker, more rigid and better crystalline than in Birdwood kaolinite. The aspect ratio of Birdwood kaolinite is therefore higher than for Georgia kaolinite which was described in [10] and estimated as 5.3 in average.

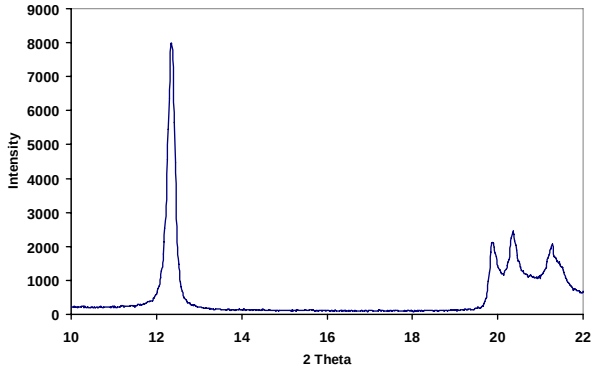

Fig. 3a XRD pattern of low and high pH Georgia Kaolinite GKa-1, powder sample, randomly oriented, sample recorded using Cu K_ radiation.

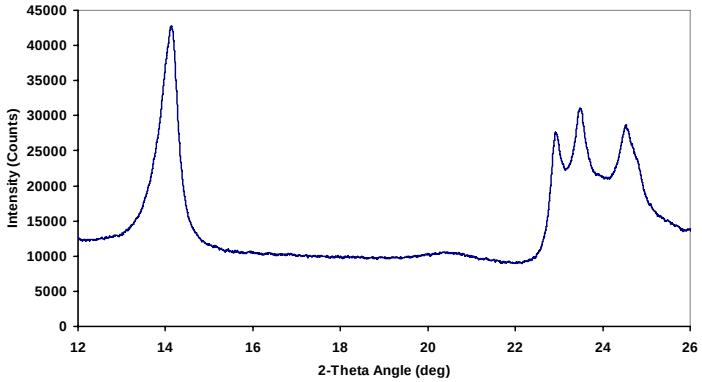

Fig 3b XRD pattern of powder sample recorded using Co K_ radiation.

The XRD pattern (Fig. 3) and other characterisations of the KGa-1b kaolinite from Georgia (USA) have been extensively studied and well-described, described in [5]. XRD of Birdwood kaolinite described in [7] displays medium crystallinity (Hinckley index 0.75) with a broad X-ray peak centred at 3.8 Å indicating that an amorphous compound may be present. A few grape shaped anatase aggregates and a few tubular halloysite particles were also observed. The amorphous material reported in [4] can occasionally been seen in SEM and AFM images. Such colloids have newer been observed in the micrographs from KGa-1 Georgia kaolinite (Fig. 2B). XRD of Georgia kaolinite proved to be better crystalline than the Birdwood kaolinite (Hinckley index 0.99). X-ray diffraction pattern of the Birdwood kaolinite has been discussed in [4] and shows a strong asymmetry on the low angle side of the 0 0 1 peak. It is suggested that interstratified smectite-like layers may be present in this kaolinite [11]. XRD of Birdwood kaolinite also displays poorly crystalline kaolinite with a broad X-ray peak indicating that an amorphous compound may be present.

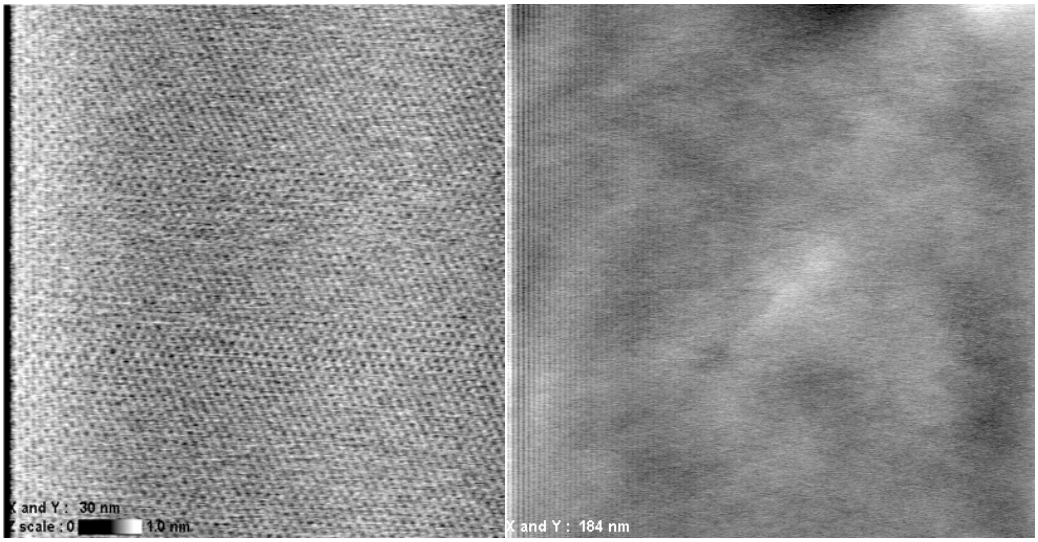

Fig 4 The high resolution AFM micrographs of the KGa-1 (left, frame is 30 nm) and Birdwood kaolinite (right, frame is 184 nm).

The AFM investigations of surface morphology of both kaolinites are presented in Fig 4 shown that KGa-1 kaolinite display clean surface where molecular arrangements of oxygen atoms in siloxane layer is clearly visible. In contrary at the surface of the Birdwood kaolinite crystal features are not visible which may be interpreted that surface in this kaolinite is rough and contaminated.

To check possible contamination Birdwood kaolinite was also studied by time of flight secondary mass spectrometry (ToFSIMS) and confirmed hydrocarbon component on top of kaolinite platelets (Fig. 5). Unfortunately it is not possible to determine how the hydrocarbon is distributed across individual kaolinite crystals faces. Some evidence for this contention has been seen in imaging of species on the aggregate surfaces. There is a clear association of CH^- fragment images with both Al+ and Si+ images possibly suggesting partially hydrophobic species but there is no evidence of any effect of these species on the electrokinetic behaviour of the kaolinite and carbonaceous contamination of surfaces is well known to be ubiquitous. If present, however, even in small concentrations, surfactants would also help to explain the stability of the micro bubbles and even gaseous film in the mineral interfaces detailed described recently in [12]. Georgia Kaolinite KGa-1 has not been investigated in ToFSIMS technique.

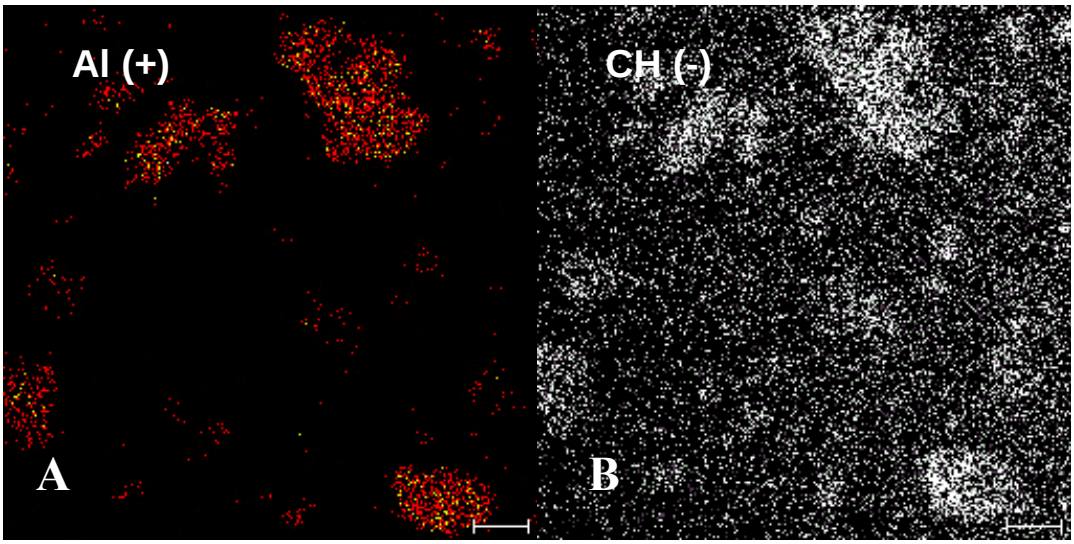

Fig. 5 TOF-SIMCE images of colloidal Birdwood kaolinite deposit on silicon wafer (scale bare 10 μm). Selected positive Aluminium ions (A) and negative ion images (B). Please note that the CH (-3) represents HC (hydrocarbons).

The grain size measurements in studied Birdwood bulk kaolin sample show that 10% of particles have diameters smaller than 0.52 μm, 50% of particles have diameters smaller than 4.62 μm and 90% of particles have diameters smaller than 20.6 μm and a specific surface area 19.7 $\pm$ 0.5 m^2g^{-1} (BET).

The Birdwood kaolinite when investigated using cryo-SEM techniques revealed edge to edge (EE) particle orientation (Fig. 6). Electron microscopy observation of the clay suspension structure shows discrete particles link together forming a coagulated spanned network (Fig. 6A) that extends throughout all the suspension via clay platelets networking (EE) orientation. This structure is similar to cellular honeycomb types described before in [13]. These chain-aggregates are few μm long and encircling large voids filled with electrolyte 1-10 μm in diameter. These highly porous super-structure build of elongated aggregates and chain associations have been described before in [9, 11, 12, 13] as a flocculated but dispersed structure. Such gel structured suspensions are typical of those observed in Birdwood kaolinite by SEM [9] are difficult to explain on the basis of DLVO theory because positively charged edges should repel each other. Furthermore, the van der Waals forces, which are the normal cause of coagulation, does not favour (EE) contacts because these would have weaker attraction than edge-to-face (EF) or face-to-face (FF) contacts.

In the high resolution cryo-SEM micrograph (Fig. 6B) however more complex particle arrangement is revealed. Colloidal in size kaolinite platelets build string like aggregate (wall of cellular structural element) but their connection is not exactly edge to edge (EE) but rather in sort of stairs-step arrangement where following platelet covers in FF arrangement, fraction of preceded particle surface. This arrangement can be only observed in the high resolution images and in lower resolution imaging it is seen as EE contact. Also platelets in such aggregates are not separate crystal in the thickness but few platelets connected in the FF arrangement with hand of card like shifted on edges. This arrangement looks suits stair step connection between aggregates and forms this long stringy like aggregate which builds the cellular structure walls.

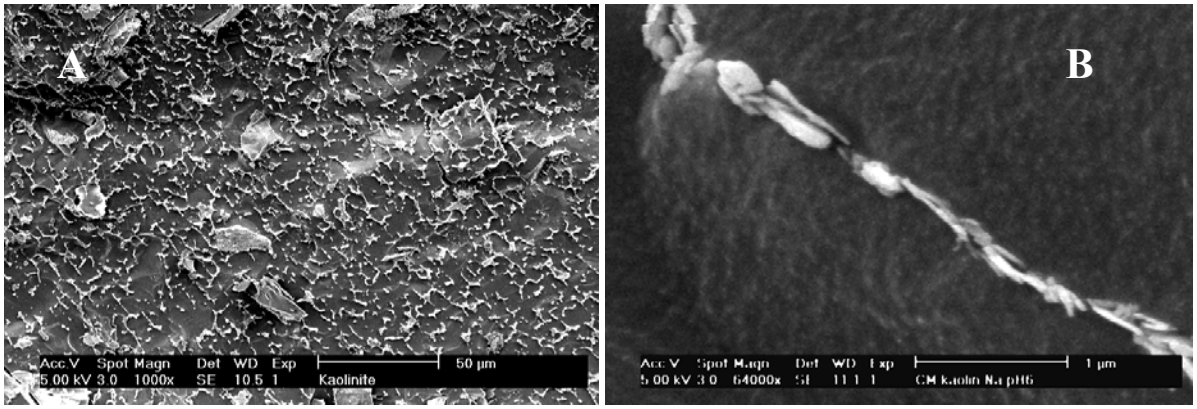

Fig. 6 Cryo-SEM micrographs of the Birdwood kaolinite in dilute aqueous suspension (4 wt%) shows in low magnification micrograph (A)- cellular texture which forms the spanned network of gelled particles in mostly edge to edge platelets and chains orientation, and in high resolution micrograph (B)- details of particle connection within chained aggregate discussed in text.

As an explanation of the predominance of such contacts it is proposed in [13] that hydrophobic like attraction between aerated edges may occur and platelets may rolling on top of each other with help of air micro-bubbles well described in [12]. Such mostly EE linked particles build long strings seen in two dimensional SEM images as sections of planar structure that connect to each other to build a spacious cell structure as shown in Fig 4B.

In the TXM stereo pare micrographs from of the 8 wt% Birdwood kaolin water suspension as shown in Fig. 7, the long chained aggregates consisted of numerous colloidal size platelets like described above in cryo-SEM investigations. These elongated chains like aggregates have been

denser packed in chaotic network in which even larger grains are immobilised. The long chained aggregates in this micrograph are well wrapped with other similar and heavily cross linked. Cellular like voids in these micrographs are usually below 1 μm in diameter. Some illusion of high packing in these micrographs can be explained by long focal length of TXM images. The long focal length (50 μm) makes many particles in thicker suspension being viewed simultaneously in focus which in effect will obscure some particles and may have negative impact on image clarity.

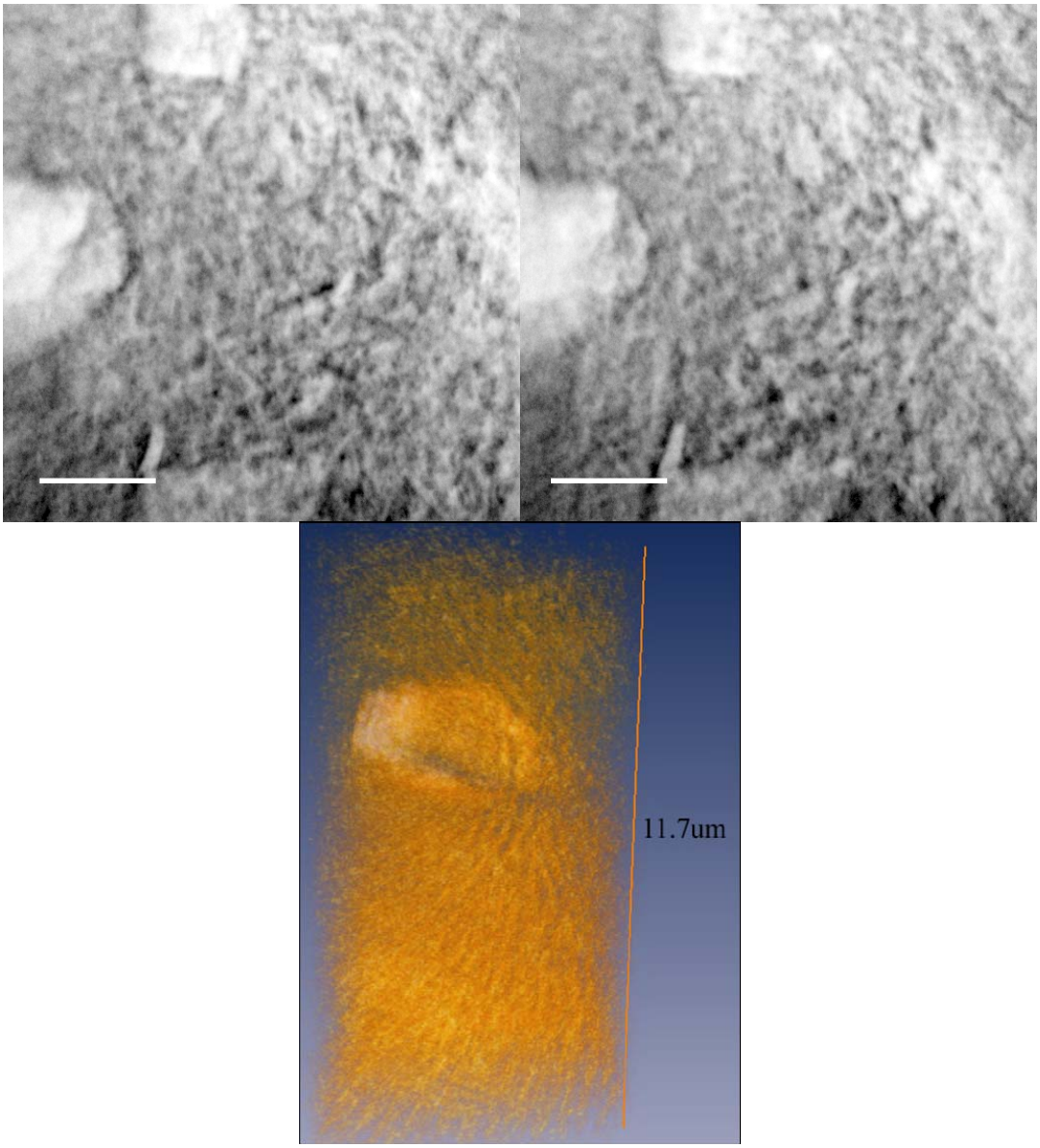

Fig. 7 TXM stereo pares micrograph (top) from water suspension in 8 wt% of Birdwood kaolin. Scale bar is 2.5 μm. (Bottom) - Snapshot from 3-D reconstruction of a Birdwood kaolinite suspension. Larger stacked crystals are entangled in the network of colloidal kaolinite platelets, characteristic string like network pattern can be observed.

271 This snapshot of 3-D stereo pares from TXM in a Birdwood kaolinite suspension demonstrated
272 in Fig. 7 show a dense network of colloidal kaolinite particles supports a larger kaolinite stacked
273 crystal of few μm in lateral dimension. This micrographs may be not giving very impressive picture
274 as compared with other well established techniques such as SEM, but enable us to examine clay
275 aggregates in 3-D through water, which has never been previously possible. Individual colloidal
276 size particles are visible in this picture magnification and distinctive string-like spongy and highly
277 oriented structure is observable when carefully studying this micrograph under stereoscope or in 3-
278 D computer reconstruction shown in [18].

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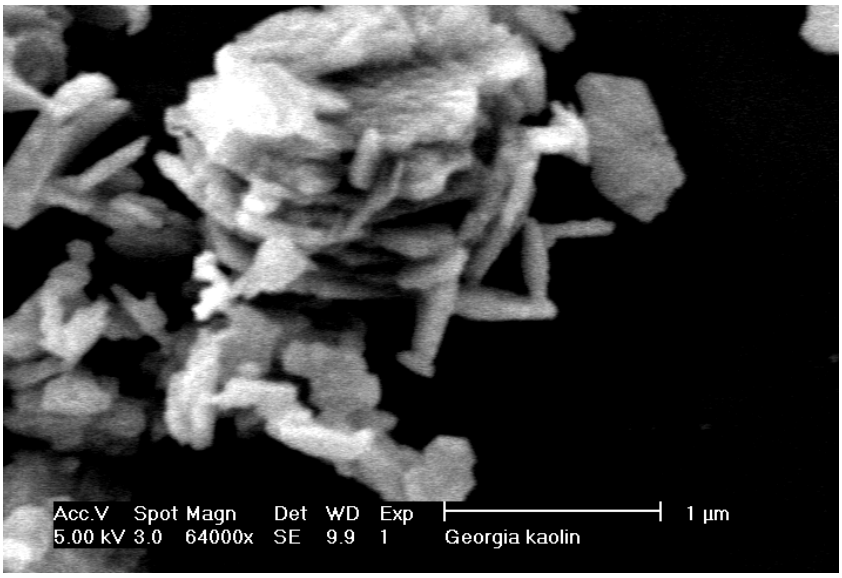


280

281 **Fig. 8 SEM micrographs of the Georgia KGa-1 kaolinite micrograph show mostly edge to**
282 **face (EF) platelet orientation.**

283

284 Studied KGa-1 kaolinite sample consists of two morphologically distinctive kaolinite
285 populations, fine to colloidal kaolinite platelets and larger stacked crystals. Ninety percent by
286 weight of the particles have an equivalent spherical diameter les than 2 μm with a median particle
287 size of 0.7 μm and a specific surface area of 15.3 $\pm$ 0.5 m^2g^{-1} (BET). The fine kaolinite fraction in
288 SEM investigations using the cryo-transfer technique (Fig. 6) show a very different particle
289 arrangement than observed in Birdwood kaolinite suspensions. Instead of EE contact arrangement
290 between platelets as in Birdwood kaolinite, in KGa-1 kaolinite most particles have edge to face
291 (EF) contacts. Such an arrangement is much less voluminous in comparison to the Birdwood

292 kaolinite suspension structure and particles in the networked flocs of KGa-1 are much closer
293 together.

294

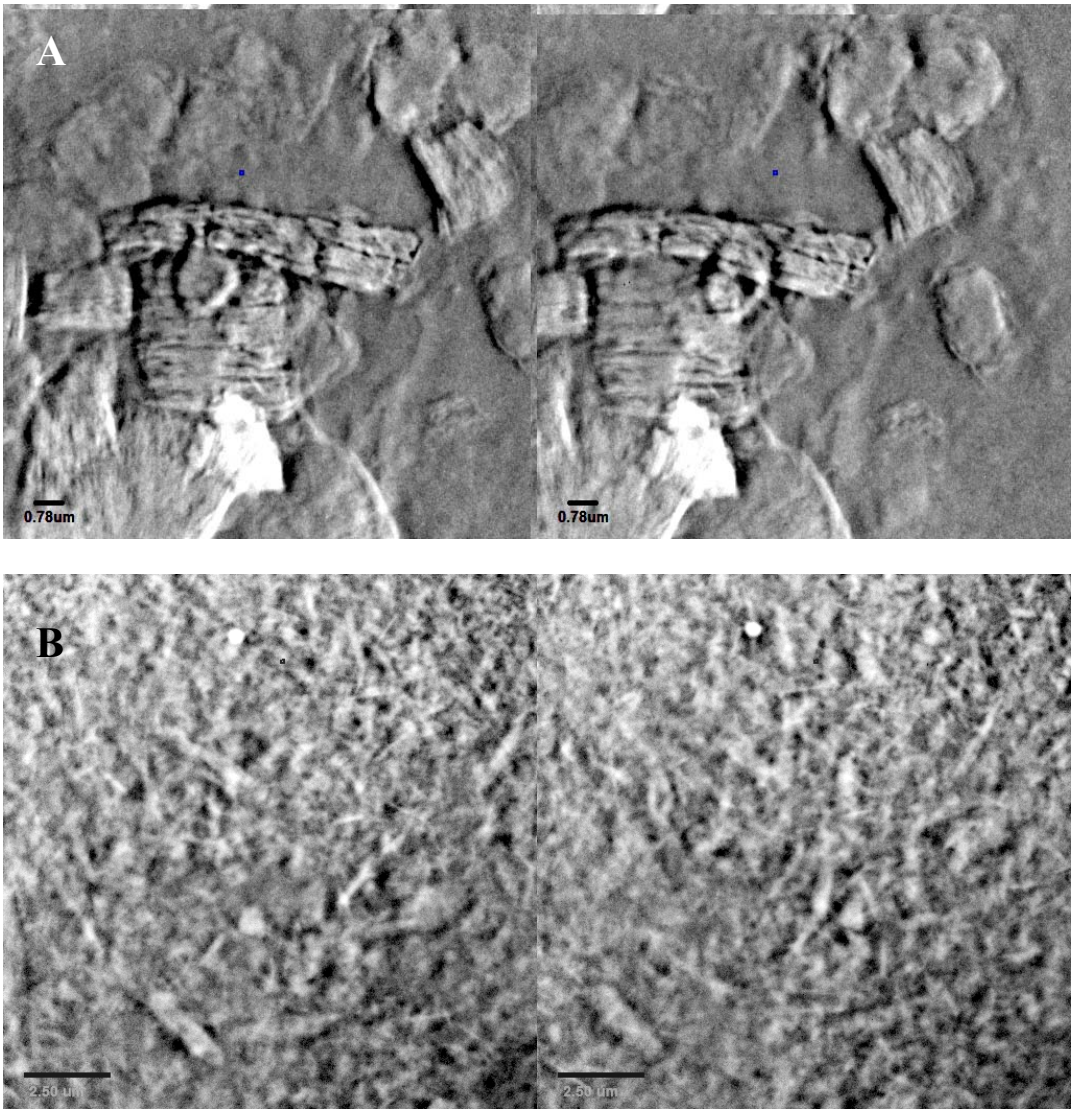
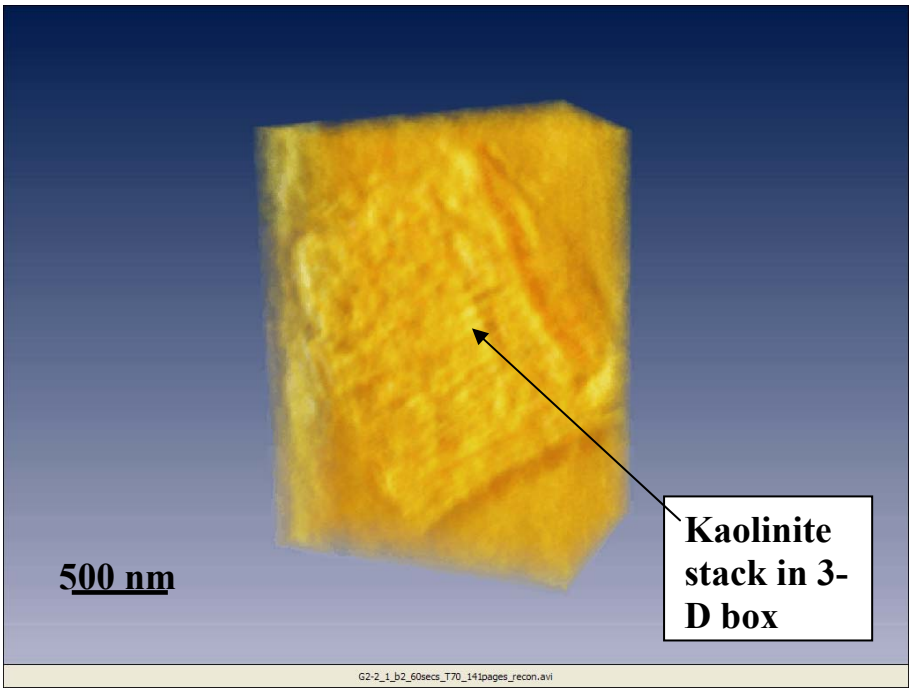


Fig. 9 TXM stereoscopic pares from KGa-1 kaolinite sample in water suspension show: (A) flock of stacked kaolinite crystals (scale bar 780 nm) and (B) mostly EF connected fine kaolinite platelets (scale bar 2.5 µm).

In sample (KGa-1) a few µm in dimension stacked kaolinite crystals, are more common then in the Birdwood kaolinite where stacks are less common and they are smaller in dimension. **In Fig. 10** two 2-D TXM micrographs of KGa-1 kaolinite shot from different angles (stereo pares) reveal aggregates consisting of randomly connected stacked kaolinite crystals 2–4 µm in dimension. Stacks show distinctive lamination 150 nm thick and randomly contact each other through rugged edges. Individual platelets up to 2 µm in diameter were observed. The aqueous solution surrounding these kaolinite stacks shows distinctive gaps between the water and solid surface at several places as well as apparent voids within aggregates. This type of gaseous bubbles and film

313 presence was detailed described in [12] where cryo-SEM investigations revealed that water not
314 wetting mineral surface leaving gaseous film on the solid/liquid interface. The gaps adjacent to the
315 mineral surface are 80–100 nm thick and appear to continuously cover substantial parts of the
316 aggregate surfaces. This gaseous gap may be because of hydrophobic interaction between siloxane
317 layer of kaolinite crystals and water. Such a gaseous blanketing at the mineral interface can be
318 eliminated by ultrasonic treatment [12]. Hydrophobic interaction between clay particles surrounded
319 in such blankets was nominated as the major factor in aggregation and structure building in [14].
320 Hydrocarbon contamination was suspected the effect of such hydrophobic interaction in Birdwood
321 kaolinite. However TXM observations recorded possible gaseous films associated with larger
322 stacks in KGa-1 kaolinite. Such nano-bubbles occurrence in area of rough edges (which are
323 common in kaolinite stacks) and in puckering between siloxane layers in talk which was described
324 in [15 and 16]. This wetting phenomenon is very complex and includes mineral/water interface
325 chemistry, contamination and morphology in nano-scale.



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327 **Fig 10 3-D reconstruction of the Kaolinite stack from coarse fraction of Georgia kaolinite**
328 **sample as seen within the aqueous solution. Stacking is visible as well as fine fraction of**
329 **kaolinite particles adhered towards stack edges which are more clearly visible when picture is**
330 **rotated. Dimension of cube is ~2×4 μm.**

331

332 In the TXM stereo pares show in Fig. 7B the colloidal fraction of kaolinite sample KGa-1 in
333 density 8 wt% is presented in 3-D space arrangement. This stereo pares as well as computer 3-D

reconstruction (not shown here) show dense suspension with particles in face to edge arrangement with most voids bellow 0.5 μm.

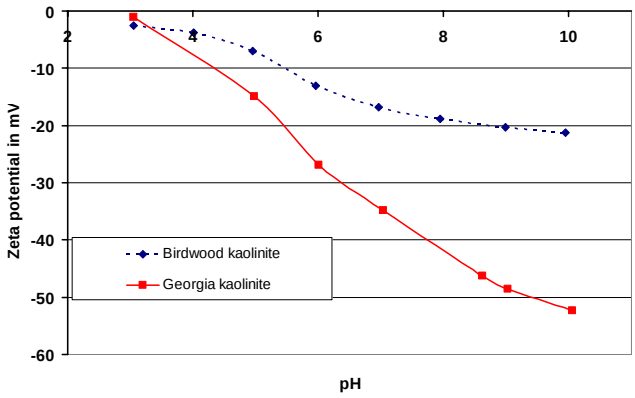

Fig. 11 Zeta potential of Birdwood and Georgia (KGa-1) kaolinite suspensions measured on AcoustoSizer.

The electrokinetic (zeta) potential measured for both kaolinite samples shown in **Fig. 11** displays major differences. The KGa-1 kaolinite sample displays typical pH-dependent profile due to OH^- groups occupied platelets edges in high pH. The Birdwood kaolinite displays smectite like, very low pH-dependence suggesting dominance of the basal plane. Such dominance may be due to blockage of particle edges by colloids, hydrocarbons and possible gaseous phase associated with colloidal hydrocarbon contamination. Such blockage of the edge sites may significantly reduce repulsive forces between particle edges and in consequence system will favourite the EE particle arrangement.

Within the context of the microscopical observations shown in Fig. 1 may be interpreted as follows: At low solids loading, separate particles and particle aggregates are suspended in the water solution and settle freely accordingly to the Stokes' law. When solid loading increases it reaches a critical particle concentration at which closest particles interact and start to form a three-dimensional structure. In the case of Birdwood kaolinite individual particles interact by attracting each other towards their edges and build an extremely large voluminous network composed of chain-like platelet assemblages. Such an extended network may fill the entire volume of a vessel. In such a case the suspension is gelled; there is no free settling in this system and compacting occurs slowly by structure recombination. The process of Birdwood kaolinite sludge recombination is described in detail in [9]. The settling velocity of thick sludges is presented also [17].

Kaolinite from Georgia (KGa-1) forms more compact aggregates by preferring edge to wall orientation and as result forms a less porous network than Birdwood kaolinite. This means that for KGa-1 kaolinite to build a gel suspension for which the gel spans the entire volume of the vessel requires a much greater solids loading than for Birdwood kaolinite (Fig. 1).

Described above differences in gelling properties in studied two kaolinite samples explained here by mayor differences in way like individual particles are connected in to tree-dimensional network may have its cause in differences in studied minerals the aspect ratio. This difference in the aspect ratio may effects in physical properties such as the lower dependability of the zeta potential in changing pH, rougher edges, and higher aspect ratio observed in Birdwood kaolinite which effects in the highly porous arrangement of individual particles in the three-dimensional network. But the mechanism influenced these differences is still obscure and need further investigations.

Conclusions

Within the context of structural differences of clay-water suspensions using the cryo-SEM and the synchrotron based TXM with 3-D reconstruction, observed in Fig. 1 behaviours can be interpreted as follows. At low solids loading, separate particles and particle aggregates are suspended in the water solution and settle freely governed by the Stokes' law. When solid loading increases it reaches a critical particle concentration at which closest particles interact and start to form a three-dimensional structure. In the case of Birdwood kaolinite individual particles interact by attracting each other towards their edges by stair step elongated aggregates and build an extremely large voluminous smectite like cellular, honeycomb type structure previously reported in [13]. Such extended network may fill the entire volume of a vessel. In such a case, the suspension is gelled; there is no free settling in this system and compaction occurs slowly by structure re-arrangement. The process of Birdwood kaolinite sludge recombination is described in detail in [9]. Because aggregates which form in the Birdwood kaolinite suspension are very long, they attracted in network building phenomenon other similar aggregates from much larger distances than dimension of single platelets. Three dimensional network build of such aggregates is extremely voluminous and suspension gels in relatively low solid content.

It is possible that hydrophobic interaction between kaolinite particles plays important role in structure making process. Possible hydrocarbon contamination at the mineral interface shown in

Birdwood kaolinite may contribute to formation gaseous microbubbles. However such film of gasses also was observed in TXM images of Georgia kaolinite interpreated as hydrophobic repelling of water by non wetted siloxane tetrahedral layer. Nature of this phenomenon is obscure and need further investigations.

Kaolinite from Georgia (KGa-1) forms compact aggregates by preferring edge to face orientations. Electrostatic interaction between particle edges and faces build network with voids diameter corresponds to particles dimensions. As the result, sediment bead is packed much denser than in Birdwood kaolinite. This means that for KGa-1 kaolinite to build a gel suspension for which the gel spans the entire volume of the vessel requires a much greater solids loading.

Observed differences in gelling properties in the two kaolinite samples may be explained in terms of major differences in particle surface chemistry and aspect ratio. These properties in the kaolinite mineral structure and possible smectite like interstratifications may effect in physical properties such as the lower zeta potential on basal surfaces, rougher face surfaces, and higher aspect ratio observed in Birdwood kaolinite which effects in the highly porous arrangement of individual particles in the three-dimensional network. But the mechanism influenced these differences is still obscure and need further investigations.

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kaolinite particles adhered towards stack edges which are more clearly visible when picture is rotated. Dimension of cube is ~2×4 μm.

Fig. 11 Zeta potential of Birdwood and Georgia (KGa-1) kaolinite suspensions measured on AcoustoSizer.