BENEFICIATION OF AN ANDRASSY AND MANSULI BENTONITE AS A DRILLING MUD MATERIAL

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Abstract

Based on the result of Geological Survey Department Malaysia investigation, the bentonite resources in Sabah is great amount. In order to reduce the overall cost in oilwell drilling and completion, the development of Sabah bentonite as drilling mud material has been initiated. In addition, Sabah bentonite is categorized as not sodium based bentonite. Appropriate beneficiation method is sought to removal some of the impurities in Sabah bentonite. There are two objectives that can be summarized in this study. The first one was to determine optimum beneficiation process. The result obtained after the beneficiation methods showed improvement in cation exchange capacity (CEC), which is about 73.33% to 90.38% increment. The higher CEC value indicates the improvement in water absorption capability of bentonite. The second objective was to determine the performance of bentonite as drilling mud material compare with standard bentonite and API (American Petroleum Institute) Specification 13A, respectively.

Keywords : bentonite, montmorillonite, beneficiation, cation exchange capacity, drilling mud material,

INTRODUCTION

Montmorillonite $\{[Al_{1.56}\ (Fe^{+3})_{0.03}\ Mg_{0.41}][Si_{3.74}\ Al_{0.26}]\ O_{10}\ (OH)_2\}(M^+)_{0.67}$ is by far the most abundant of the smectite clay minerals. It is the predominant mineral composing bentonite as well as fuller's earths throughout the world and is major component of soils formed in arid and semiarid areas. In drilling industry, montmorillonite is generally classified as sodium (Na) or calcium (Ca) types, depending on dominant exchangeable ion. The major problems facing the beneficiation of Sabah bentonite is their low concentration of smectite, high level of iron contaminant and inconsistent composition. Previous studies on some Sabah bentonite suggested that, without beneficiation, they were unsuitable for drilling mud material. Response of this bentonite to Na-exchange was poor and their hydration, plastic and rheologycal properties were

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inferior to those commercial bentonite [7]]. Several attempts were made to upgrade to meet OCMA/API specification for drilling fluid and other industrial uses. [1,4,9]. This paper presents optimum beneficiation process for upgrading andrassy and mansuli bentonite and their application in petroleum industry. The tests were conducted on run-of-mine (ROM) and chemically activated samples. Chemical methods involve extracting of the mineral with organic acid also cation exchange capacity measurement.

EXPERIMENTAL WORK

Bentonite sample

In this study, two groups of local bentonite samples were collected, namely SA5-1, SA5-3, SA5-4 and SA5-7 (N4^{0}18.97'- E 117^{0}57.37') from Andrassy area in Tawau district and M4 (N5^{0}7.35'- E118^0 12.03') from Mansuli area in Lahad Datu district. The SA5-1and SA5-3 sample were collected exactly at 0.5 m depths, SA5-4 and SA5-7 sample was collected at 1.0 – 1.5 m depths and M4 at 0.3 m depths. The field sampling from Mansuli area were taken mainly in area underlain by the Ayer Formation, which collectively form the Segama Group and is interpreted to be Miocene in age The Andrassy area is underlain mainly by the high level of alluvium and volcanic rock, and occur in a bed underlying by Pleistocene to Holocene in age [8].

Experimental procedure

Beneficiation process

Bentonite samples from the field will be dried in the oven at 35 °C for four hours until reach moisture content less than 10%. Then the sample will be crushed using a Jaw crusher to 100% below 5 mm in size. This was followed by aero cyclone model EPC100P [5], to disintegrate the smectite from its associating gangue minerals. Both the aero cyclone underflow (coarser than 63 μm) and the overflow fractions (finer than 63 μm) were filtered dried at 55°C and weighed. Underflow fractions, which were heavily contaminated with iron and free-silica impurities, were

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discarded. Meanwhile, the overflow fractions were used as bentonite pre-concentrates. The latter products were further upgraded by extracting, their residual iron impurities, with 7.1 kg m^{-3} oxalic acid solutions for 2 h with 80 °C temperature. The bentonite was beneficiated to improve its smectite content by removing the associated gangue minerals (mainly iron and free-silica).

Physical and Chemical Properties Determination

Common chemical property of montmorillonite is pH, cation exchange capacity (CEC) and specific surface area (SSA). pH value were measured by pH meter Model Hanna H18424, which adapted generally from the BS1377:Part3: 1900:9. SSA was measured using methylene blue spot test [6]. In addition, the physical properties determinations include the Atterberg limit such as liquid limit (LL), plastic limit (PL) and plasticity index (PI), moisture absorption (MA), moisture content (MC) and ignition loss (IL). Methylene Blue Test (MBT) is used to estimate the CEC. A sodium-based bentonite should have a CEC value same as that of montmorillonite (80 –10 meq/100 grams). Approximately 1 gram of montmorillonite sample will be tested in 50 ml of distilled with about 0.5 ml of 5 N sulfuric acid added. The montmorillonite solution will than boiled gently for 10 minutes. The CEC is measured by conductomercial titration after cation exchange, meq/100grams. In addition, the chemical composition of montmorillonite will be tested by EDAX Philip Series-40 instrument. This equipment can calculated quantitatively of bentonite element based on the emission of electron in it's orbital. The physical study values are used to obtain information on the nature and quality of the mineral by using Atterberg Limits Test, such as plastic limit (PL), liquid limit (LL) and plasticity index (PI). The standard testing method for liquid and plastic limit according to British Standard (BS) specification is BS1377 Part2 1990. 4.3 and BS1377 Part2 1990. 5.3. respectively. Liquid limit and plastic limit also refer as Atterberg limit, which depending on the moisture content of sample. The liquid limit provides the moisture content at which the clay changes from plastic to the liquid state. While the plastic limit was

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simply the moisture content at which a ball of clay when rolled to a diameter of 1/8 inch. On the other hand, plasticity index is the difference between liquid limit and plastic limit. In addition, the moisture adsorption (MA), moisture content (MC) and ignition loss (IL) of sample will also be determined since the qualitative mineral content of sample can be studied. Moisture adsorption (MA) is defined as the percentage of water lost when clay from a saturated atmosphere (around 20°C) is dried in an oven at 105°C. The analysis of moisture content followed the BS1377 Part2: 1990. 3.2. Moisture adsorption (MA) value can be used to predict the mineralogy nature for clay. Moisture content (MC) is the percentage of water lost when clay from normal room temperature atmosphere (at around 20°C) is dried at 105°C. While the ignition loss is the percentage of weight lost when a dried clay (at 105°C) is fired to 1000°C in furnace.

Surface area of each sample was determined using a technique by [6]. Cation exchange capacity (CEC) of the samples was determined by a methylene blue absorption technique (BS 1337, part 2, 1990). Swelling indices were determined by a standard technique of measuring the gel volume formed from the addition of 1 g, air-dried, sample in 10 ml of water after 24 h when the last portion of the material was introduced into the cylinder.

Rheological tests

In testing the rheological properties of the activated products, the suspension were re-agitated for 5 min, using a mixer, and then transferred to the viscometer at a rotor speed of 600 rpm. The readings of the viscometer were recorded at 5 min intervals until a stable reading was attained. This procedure was repeated at a rotor speed of 300 rpm. The reading of the viscometer at 600 rpm is taken as the apparent viscosity (A.V.) while the difference between the readings taken at 600 and 300 rpm represents the plastic viscosity (P.V.). The yield point (Y.P.) is calculated from the difference between the reading of viscometer at 300 rpm and plastic viscosity. Fig. 1 shows a simplified flow sheet for the beneficiation scheme. Testing of the samples as a raw material for drilling fluid was carried out according to Section 11 of the API specifications

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(American Petroleum Institute, 1993).

RESULTS AND DISCUSSION

The Mineralogy

After organic acid beneficiation, the mineral composition of the Andrassy and Mansuli samples (see Table 1 and Table 2) changed with SA5-4 sample has 49.35% improvement and M4 samples 67.71% increment, respectively. From quantitative analysis, the overall montmorillonite composition in the bentonite slightly changed. It is clear that the beneficiation by using oxalic acid had successfully removed iron content as impurities in the sample and the quantity of the montmorillonite in the all sample except SA5-7 are improved.

Physical Properties

MC, MA, IL

The MC, MA and IL results of beneficiated Andrassy and Mansuli are as shown in Table 3. It is observed that the MC for the beneficiated sample had been increased in the range of 9.92 % to 63.33 % of the original values. The beneficiated M4 sample from Mansuli area, had been dramatically increased i.e., 63.33 % of the original value because of reduced free-silica content. It is indicated that the beneficiated SA5-3 sample has a very low MC as compared to other bentonite samples due to low water adsorption and cation exchange since its closer to plastic kaolin region. The water in the lattice of bentonite mineral is important as an agent of chemical reaction such as ion exchange and adsorption into the mineral. The mineral moisture distribution is important for the sorption characteristics because mineral with higher specific gravity are transported in solution. The reference bentonite sample shows higher moisture content and plasticity index compared to beneficiated Andrassy and Mansuli samples. The low moisture content in Andrassy and Mansuli samples was due to the low percentage of montmorillonite mineral as shown in Table 1 & Table 2. There are some improvements in MA values as shown in Table 3. due to reducing iron and free-silica content in the samples, i.e. increment of 3.42 % to

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79.26% which is still not as good as the reference bentonite. The IL values Andrassy and Mansuli samples are higher than raw bentonite sample, except for SA5-3 sample. Since the water molecules in SA5-3 sample is located within the tetrahedral and octahedral sheets of bentonite crystal structure.

Atterberg Limit

Generally, after beneficiation Andrassy samples show improvement in liquid limit with increment varying from 89.82 % to 229.63 % of the original values, as shown in Table 4. These samples also showed improvement in plastic limits, with increment varying from 0.40 % to 37.13 %. This again proved that the montmorillonite mineral plays an important role that influences the plasticity of a sample. These results show that the iron and free-silica content as impurities in the raw bentonite decreased, but the improvement still not as good as reference bentonite.

As shown in Table 4, after beneficiation, the plasticity index of beneficiated Andrassy and Mansuli sample had also been improved with increment varying from 181.78% and 582.57 %. This means that the water absorption capabilities of these samples are better than those raw one. The low percentage of bentonite mineral in the sample has resulted in lower moisture content and plasticity index.

Chemical Properties

CEC

From the Table 5, we can see that the CEC of beneficiated Andrassy and Mansuli samples had been improved. This improvement indicates that the calcium ions on the Andrassy sample unit layers had been replaced by sodium ions. This will have a positive impact to the hydration and swelling capability when used as a material in drilling mud. However, their CEC values still much less than the reference bentonite (CEC of 80 meq/100 g).

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Chemical Composition
After beneficiation, the results of chemical composition generally indicated that the Si and Al contents of these samples had been substituted by Fe and this kind of phenomenon is generally known as "isomorphous substitution". Isomorphous substitution describes the replacement of Al or Si ions inside the interior of the bentonite particle by ions of lower positive valency, such as Fe. Since the substitution occurs on the interior of the particle, the charge deficiency is manifest over the entire surface of the bentonite particle, leaving a net negative charge on the particle. Sometimes, ions other than Al (such as Fe and Mg) also can replace some of the Si ions in the substitution process. This isomorphous substitution is the primary source of charge of bentonite and this net negative charge will attract the interlayer material such as water molecules and cationic materials.

Specific Surface Area
Specific surface area are shown in Table 7. It is clearly seen that the beneficiated Andrassy samples show some improvement in specific surface area. The treatment with oxalic acid had successfully remove an iron in the sample therefore the specific surface area of all sample improved. This proved that the SA5-4 sample (640 m^2/g) is the most satisfies sample that has a specific surface area closed to the reference bentonite (660.37 m^2/g). However, it is still has insufficient absorption capability since the specific surface area still not to compared to the reference bentonite due to its low content of montmorillonite mineral in the local sample. The SA5-1, SA5-7 and M4 samples also exhibit increment in specific surface area. Since there is only limited or small amount of montmorillonite mineral, these beneficiated samples still failed to reach the specific surface area of reference bentonite (660.37 m^2/g). As for SA5-3 sample, due to its low

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content of montmorillonite mineral an increment of specific surface area of 17 % is considered encouraging.

Drilling Mud Performance

As shown in Table 8, the beneficiated Andrassy and Mansuli sample still failed to meet the API specification 13A except for YP/PV ratio and moisture content. However, it is clearly seen that the beneficiated samples show improvement in two important parameters; viscometer 600 rpm dial reading and filtrate loss, i.e., around 380 % to 700 % of viscometer 600 rpm dial reading and, 60% to 85 % of fluid loss reduction. This generally caused by not all impurities had been removed from the sample and less amount of montmorillonite content for the local bentonite samples, especially the SA5-3 sample. Hence, it is desirable to find a solution and the possible solution is to add some polymer extender that can improve the viscosity generated.

CONCLUSION

The bentonite beneficiated based on organic acid treatment can be achieved by applying at organic acid concentration of 7.1 Kg m^{-3}, pH less than 2, 80°C temperature and 2 hours stirring time. It was found that the highest CEC is 72 meq/100 g from original value of 41 meq/ 100 g. In addition, Sabah bentonite can not meet API Spec 13A of drilling mud material. Otherwise, the beneficiated bentonite performance as drilling mud material can be improved by 3% wt Tannathin addition.

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Table 1. Semi-quantitative analysis of beneficiated Andrassy and Mansuli samples

Mineral Composition	Reference Bentonite	Beneficiated Bentonite Samples				
		SA5-1	SA5-3	SA5-4	SA5-7	M4
Montmorillonite	****	**	*	*	**	*
Quartz (SiO_2)	nd	*	*	*	*	*
Kaolinite (($Al_2Si_2O_5(OH)_4$)	nd	*	*	*	*	tr
Illite (($(K, H_3O)(Al, Mg,Fe)_2(Si,Al)_4O_{10}$)	nd	*	*	*	*	*
Others	* Feldspar ($KAlSi_3O_8$)	* feldspar	tr feldspar	tr feldspar	nd	* feldspar

Keys: ****: dominant, ***: major, **: minor, *: appreciable, tr: trace, nd:not detect

Table 2. Quantitative analysis of beneficiated Andrassy and Mansuli samples

	SA5-1	SA5-3	SA5-4	SA5-7	M4
Montmorillonite content (%Volume)	14.27	9.63	13.89	10.59	14.16
Percentage of change after beneficiation process (%)	18.94	2.96	49.35	-28.45	67.71

Table 3. MC, MA and IL values of beneficiated Andrassy and Mansuli samples

Bentonite Sample	MC		MA		IL	
	%	% of change	%	% of change	%	% of change
Reference Bentonite	16.732	-	24.51	-	5.43	-
SA5-1	12.58	33.97	21.35	79.26	11.45	10.95
SA5-3	11.99	0.42	17.62	0.46	11.25	-0.18
SA5-4	12.65	33.30	22.85	6.13	11.45	1.24
SA5-7	14.85	9.92	22.98	3.42	7.52	3.72
M4	12.07	63.33	16.4	72.45	10.287	10.49

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Table 4. The Atterberg limit of beneficiated bentonite samples

Bentonite	Liquid Limit (%)		Plastic Limit (%)		Plasticity Index (%)	
Sample	%	%change	%	%change	%	%change
Reference Bentonite	700	-	65	-	635	-
SA5-1	259.8	165.62	55.75	6.17	204.05	266.01
SA5-3	120.25	89.82	32.25	0.40	88	181.78
SA5-4	278.5	126.72	49.25	5.57	229.25	365.48
SA5-7	458.98	134.05	58.65	15	400.33	582.57
M4	267	229.63	40.85	37.13	226	341.32

Table 5. CEC of beneficiated bentonite sample

Bentonite Sample	CEC (meq / 100 g)	
		% of change
Reference Bentonite	80	-
SA5-1	65	58.54
SA5-3	60	22.45
SA5-4	72	33.33
SA5-7	68	44.68
M4	36.05	50.21

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Table 6. Chemical composition of beneficiated Andrassy and Mansuli samples

Chemical Element	Chemical Composition (% weight)										
	Reference Bentonite	SA5-1		SA5-3		SA5-4		SA5-7		M4	
			% of change		% of change		% of change		% of change		% of change
O	38.24	44.57	22.92	38.53	4.79	42.64	17.63	38.42	-4.55	34.46	-2.68
Na	2.21	1.18	103.45	0.36	-14.29	0.74	17.46	0.83	-22.43	0.3	-67.74
Mg	2.23	1.35	-14.56	1.57	-6.55	1.72	1.18	1.9	4.76	1.48	-10.30
Al	11.68	12.64	-6.37	13.66	0.74	12.04	-14.49	13.37	-0.59	13.76	43.48
Si	35.06	29.76	-9.32	32.3	-1.28	29.26	-13.79	32.25	3.66	32.47	12.94
P	1.31	0.47	-18.97	1.35	-25.82	4.43	129.53	1.8	9.09	1.32	-69.37
Cl	0.38	nd	-	0.46	-99.54	0.17	-79.76	0.9	190.32	0.48	9.09
K	0.67	0.98	-39.13	1.03	-12.71	1.01	-18.55	1.41	24.78	1.18	162.22
Ca	1.69	1.36	-16.05	1.52	-15.56	1.38	17.95	1.86	28.28	1.51	27.97
Ti	0.44	0.53	-50.47	1.28	-3.76	0.53	-25.35	0.75	-17.58	1.37	149.09
Mn	0.68	0.47	51.61	0.91	21.33	0.26	-27.78	0.64	88.24	0.96	269.23
Fe	5.41	5.96	-22.09	7.04	-10.89	5.82	-18.60	5.85	-10.28	8.71	-47.18
Total	**100**	**99.27**		**99.37**		**100**		**99.98**		**98**	

Table 7. Specific surface area of beneficiated Andrassy and Mansuli samples

Bentonite Sample	Specific Surface (m^2/g)	
		% of change
Reference Bentonite	660.37	-
SA5-1	540	70.35
SA5-3	340	17
SA5-4	640	47.13
SA5-7	400	36.52
M4	300	104.50

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Table 8. Suspension properties of beneficiated Andrassy and Mansuli sample

Polymer Dispersant		Viscometer dial reading		Gel (lb/100ft^2)		pH	Filtrate (ml)	PV (cP)	YP (lb/100ft^2)	YP/PV
		300 rpm	600 rpm	10second	10minute					
SA5-1										
Tannathin	1.00%	4	7	0	0	8.5	18	3	1	0.3
(% wt)	2.00%	6	11	0	0	8.5	15	5	1	0.2
	4.00%	10	15	0	0.5	8.5	14.5	5	5	1
	1.00%	3.5	6	1	17	8.5	32	2.5	1	0.4
	2.00%	7.5	12	0	0	8.5	24	4.5	3	0.7
CMC (% wt)	4.00%	10.5	16	0	0	8.5	20	5.5	5	0.9
SA5-3										
Tannathin	1.00%	6	10	0	0	8.5	15	4	2	0.5
(% wt)	2.00%	10	15	0	0	8.5	14	5	5	1
	4.00%	11	18	0	0	8.5	12	7	4	0.6
	1.00%	9.5	15	4	20	8.5	32	5.5	4	0.7
	2.00%	16.5	23	7	35	8.5	20	6.5	10	1.5
CMC (% wt)	4.00%	16	25	8	45	8.5	17	9	7	0.8
SA5-4										
Tannathin	1.00%	7.5	12	0	8	8.5	14	4.5	3	0.7
(% wt)	2.00%	16	23	3	12	8.5	12	7	9	1.3
	4.00%	9	14	2	10	8.5	10	5	4	0.8
	1.00%	14	19	8	36	8.5	28	5	9	1.8
	2.00%	21	28	12	38	8.5	14	7	14	2
CMC (% wt)	4.00%	19	30	15	40	8.5	15	11	8	0.7
SA5-7										
Tannathin	1.00%	6	9	0	9	8.5	14	3	3	3
(% wt)	2.00%	10	15	2	12	8.5	11	5	5	5
	4.00%	7	13	2	9	8.5	9	6	6	1
	1.00%	5.5	8	7	17	8.5	25	2.5	2.5	3
	2.00%	7.5	10	11	25	8.5	20	2.5	2.5	5
CMC (% wt)	4.00%	11	18	8	45	8.5	15	7	7	4
M4										
Tannathin	1.00%	3	5.5	3	4	8.5	19	2.5	0.5	0.2
(% wt)	2.00%	8	13	2	3	8.5	18	5	3	0.6
	4.00%	5	10	0	0	8.5	15	5	0	0
	1.00%	2	4	6.5	9	8.5	45	2	0	0
	2.00%	4	6	0	0	8.5	35	2	2	1
CMC (% wt)	4.00%	5	8	0	0	8.5	25	2	2	0.7

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