Preparation Iraqi Attapulgite Clay Using Wet Sieving Process to Be Suitable for Oil Wells Drilling Fluid

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Abstract

Wet sieving is one of active separation process for clay mineral. In this work, wet sieving was applied to separate attapulgite clay from quartz for Iraqi formation to make it suitable in drilling fluid preparation. The Influence of wet sieving conditions (stirring speed, stirring time and mesh size) on quality and quantity properties of attapulgite clay were investigated. Three stirring speeds (850, 1300 and 1800 rpm) at three stirring times (5, 15 and 20min.) were tested in wet milling process, in addition to three mesh sizes (20,38 and 75µm) were applied in wet sieving process. XRD and FE-SEM were used for mineralogical and morphological characterizing for attapulgite clay. Rheological properties and yield of clay were tested at fresh and salt water using ofite viscometer. Yield of the process was measured by weight differences of clay before wet sieving and after purification process. The results showed that the stirring speed and stirring time had great action on raising the yield of process, where higher yields of process was obtained at 1800 rpm stirring speed for 20 min. at mesh size 75 µm. the results also showed, the mesh size had Direct proportion with a yield of process, while it had a revers fit with dispersion of attapulgite clay. However, best apparent viscosity (15.83 and12.06 Cp) for fresh and salt water respectively, and higher yield of clay (94.21and 81.71 barrel/ton) for fresh and salt water were gained at sieving with 38 µm mesh size. Final result, the best wet sieving condition were stirring speed1800 rpm for 20 min. at 38 µm mesh size.

Keywords: Iraqi attapulgite clay, Wet sieving process, Rheological properties, Stirring speed, Mesh size.

1- Introduction

Attapulgite is hydrated magnesium aluminum silicate mineral, attapulgite be made up of blocks and channels "ribbon-like" sheets lengthening in the c-axis direction [1, 2]. These elongate crystals (also called crystal bundles) are inactive, non-swelling, form a random lattice having the ability to trap water and providing unique thickening, suspending and gelling properties [2]. These qualitative properties are qualifying attapulgite clay to use in oil wells drilling fluid [3, 4].

Rheological properties of drilling fluid are affected with two groups of factors hydrodynamic and non-hydrodynamic. Hydrodynamic effects are the interactions between dispersion fluid and the solid particles with size larger than 10µm, which influenced by particles concentration and their shape, size and size distribution and the rheology of suspending fluid. The non-hydrodynamic effects which consist the colloidal properties of dispersions (particles size <2 µm) where the attractive forces between clay particles are so strong for that a rigid network is obtained [5].

However, hydrodynamic and non-hydrodynamic factors are affected by the pretreatment conditions such as stirring shear rate, stirring time, type of stirrer and temperature etc. [6]. Pretreatment can be defined as a set of operations were carried out during attapulgite clay preparation, where these operations are aimed to tackle productivity and economic issues of preparation process in addition to enhance quality of produced attapulgite.

Many researchers were studied the influence of pretreatment condition on attapulgite properties, for instance the impact of acidic treatment conditions (acid type and acid concentration) on attapulgite adsorption capacity were studied by Zhifang Zhang et al.2016[7]. Influence of acid concentration on rheological properties of attapulgite was studied by Feng-shan Zhou et al.2015[8]. The impact of grinding time on crystal structure and amorphous degree of attapulgite clay were investigated by Jin et al. 2004[9]. The impact of stirring conditions (stirring speed and stirring time) on the flow curves and rheological properties of clay–water dispersions was studied by C. Viseras et al. 1999[6].

Generally, process conditions are realized with many factors such as nature of clay ore, required quality and quantity and other technical factors, therefore; fixing process condition is not an easy matter.

Rare studies have relied on the qualitative and quantitative properties of attapulgite clay in determining the conditions of preparation processes. This work was devoted to study the impact of wet sieving conditions on quality (rheological properties, yield of clay) and quantity (yield of a process) of Iraqi attapulgite clay to be used in oil wells drilling fluid. Furthermore, sitting wet sieving process layout based on the balance between the quality and quantity of the produced attapulgite clay.

2- Experimental Work

2.1 Materials

Selective samples of attapulgite rock were collected from Bahar AL Najaf region at meddle part of Iraq. The mineral analysis of the rocks is shown in Table 1. Samples were crushed by using Retch jaw crusher machine (type BB200 rostfrei – Germany) to obtain small cubic particles with size ≈ $1cm^3$.

Table .1 the mineral analysis for attapulgite rock

Minerals	Calcite	Quartz	Attapulgite	Illite	Halite	Chlorite	Albite
%	37.8	25.7	9.9	8.3	3	6.3	9.1

2-2 Sample Preparation

2-2-1 Wet Milling Process

Crushed attapulgite ore (≤1cm3) was wet milled at solid / liquid ratio 1 to10 w/v by using Heidolph electrical blender and full nutrition broken machine production engineering and metallurgy department/ university of technology. The prepared suspension was taken and separated from quartz by wet sieving process.

2-2-2 Purification Process

Purification process for sieved attapulgite was devoted to remove calcite mineral. This process was implemented as following sequence, first, adding sieved attapulgite clay to 1.138 M acetic acid at liquid / solid ratio 10 to1 v/w in glass container under mechanical stirring (800 rpm) at 28˚C for 1 hour. Second, washing with distilled water until pH values be 7. Third, wet sieving process was applied at the studding mesh size. Finally, the achieved attapulgite clay was dried at 105°C for (3hour).

2-2-3 Yield Measurement.

Wet sieving is an active separation process for clay mineral. Wet milling conditions were fixed based on the yield (productivity) of wet sieving process, where the yield of process was measured by taking the weight before wet sieving (input) and after purification process (output), and then applied eqation1. Operating condition such as stirring speeds, stirring time and mesh size were tested. However, three stirring speeds (850,1300 and 1800 rpm) were applied at three stirring times (5,15and 20 minutes). The produced suspension was wet sieved at different mesh sizes (20,38 and 75 µm) then dried by Binder drying oven at 105°C for 3 hours. Then the yield of process was measured. All results of the above work were repeated for three times and the average was adopted. Note Heidolph electrical blender was used at stirring speed 850 and1300 rpm, while full nutrition broken machine used at stirring speed 1800rpm.

- Yield of process $=\frac{output}{input} *100$ ……. (1)

2.2.4 Mineral TEST

X-Ray diffraction test was implemented by using SHIMADZU X-ray - 6000Diffractometer at Nanotechnology and Advanced Materials Research Center, University of Technology– Iraq. ICDD's PDF-4+ - Phase Identification & Quantitation was used to determine quality and quantity of resulted minerals from wet sieving and purification processes.

Purified Attapulgite samples were characterized by HITACHI S-4160 Field Emission Scanning Electron Microscope at the electrical engineering department, Tehran University- I.R.Iran.

2.3.4 Rheological Properties Measurements

Rheological properties (apparent viscosity) and yield of clay were taken as qualitative properties for purified Iraqi attapulgite clay. In this work, the impact of mesh size (mesh number) on the quality of attapulgite clay was tested. Where, sieved attapulgite clay at different mesh size 20, 38 and 75 µm were used as viscosifier in water base drilling fluid. Nevertheless, the basic drilling fluid was prepared according to American petroleum institute (API) standard, where 22.5g of purified attapulgite clay was mixed with 350 ml water (distilled and brine) using Waring Blender at low speed for 20 min. After blending, the drilling fluid was left to rest for 18 hours at room temperature. Later; Drilling fluid was remixed at low stirring speed for 5 minutes before measuring rheological properties (apparent viscosity, plastic viscosity, yield point and gel strength) by using Ofite viscometer model 900 with 12 speed. The API standard formulas were applied to calculate the rheological properties of drilling fluid [10]. The test was repeated three times and the average was adopted.

Apparent viscosity (AV) $= 0.5\ \theta 600$ mpa. s …(2)

Plastic viscosity (pv) $= \theta 600 - \theta 300$ mpa. s …(3)

Yield Point (YP) $= 0.511(\theta 300 - pv)$ pa …(4)

2.2.5Yield of clay measurement

The yield of clay for the prepared drilling fluid was identified by measuring apparent viscosity for fresh and salty water at different clay content (3, 6, 9 and 12 wt. %). The relationship between clay content (wt. %) and apparent viscosity (cp) was sketched. The intersection point between apparent viscosity at 15 cp and clay content represents the x value; which will be used in Eq. 5 to calculate the yield of clay [11].

$$\text{Yield of clay} = \frac{572}{x} - 3.4 \qquad \text{.... (5)}$$

2.4.4 Stability (sedimentation) measurement

The stability of sieved attapulgite clay at different mesh sizes (20,38 and 75µm) was measured by dispersing 2 g of clay in 120 ml distilled water with waring blender at low stirring speed for 20 minutes, then the dispersing fluid was poured into the 100 ml measuring cylinder and allowed to settle for 100 h. The location of the solid/liquid boundary was recorded at fixed periods of time

3- Result and Discussion

3.1 XRD analysis

Figure 1 shows XRD patterns of attapulgite clay before and after acidic treatment with 1.138 M acetic acid. The peak at (2θ= 8.31˚) identify attapulgite clay, where the peaks at 2θ = 20.85° and 26.63°are the characteristic peaks of quartz impurity. XRD test shows the characteristic peaks of calcite mineral at 2θ = 29.49°is disappeared by acetic acid treatment.

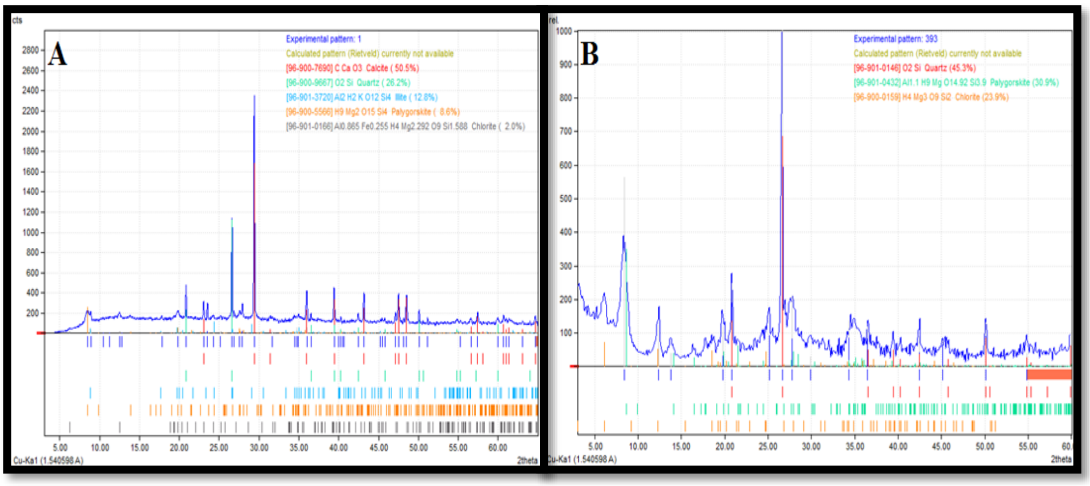

Figure1.The XRD pattern a) purified clay b) wet sieved clay.

3-2 Yield Results

The results of yield measurement are shown in figure 2, the figure shows many points, first it can clearly see the direct proportion relationship between yield of wet sieving process and both of stirring time and stirring speed. However, the main purposes of milling process are size reduction and increase liberation of target ore from rock mineral [12,13]. In this work, shear rate is increased proportionally with stirring speed leading to increasing in attapulgite erosion rate, by other meaning increasing liberation of attapulgite clay. Stirring time has the same impact of stirring speed on erosion rate of attapulgite clay. Thus, yield of wet sieving was increased [6].

Second the yield of wet sieving process reaches to a study and maximum value (44.28%) after 5min. of wet milling process at 1800 rpm stirring speed and mesh size 75µm. This result could be due to the nature of attapulgite rocks in Bahar Al-najaf region as sedimentary formation created by the sediments of the Euphrates River [14].

Third at all sizes, there is a wide gap in yield of process between stirring speed 1300 rpm and 1800rpm. This could be relate to deference in milling impeller design for two milling devises (Heidolph electrical blender and full nutrition broken machine), where the full nutrition broken machine was more efficient than Heidolph electrical blender to liberate attapulgite clay from quartz within attapulgite rock.

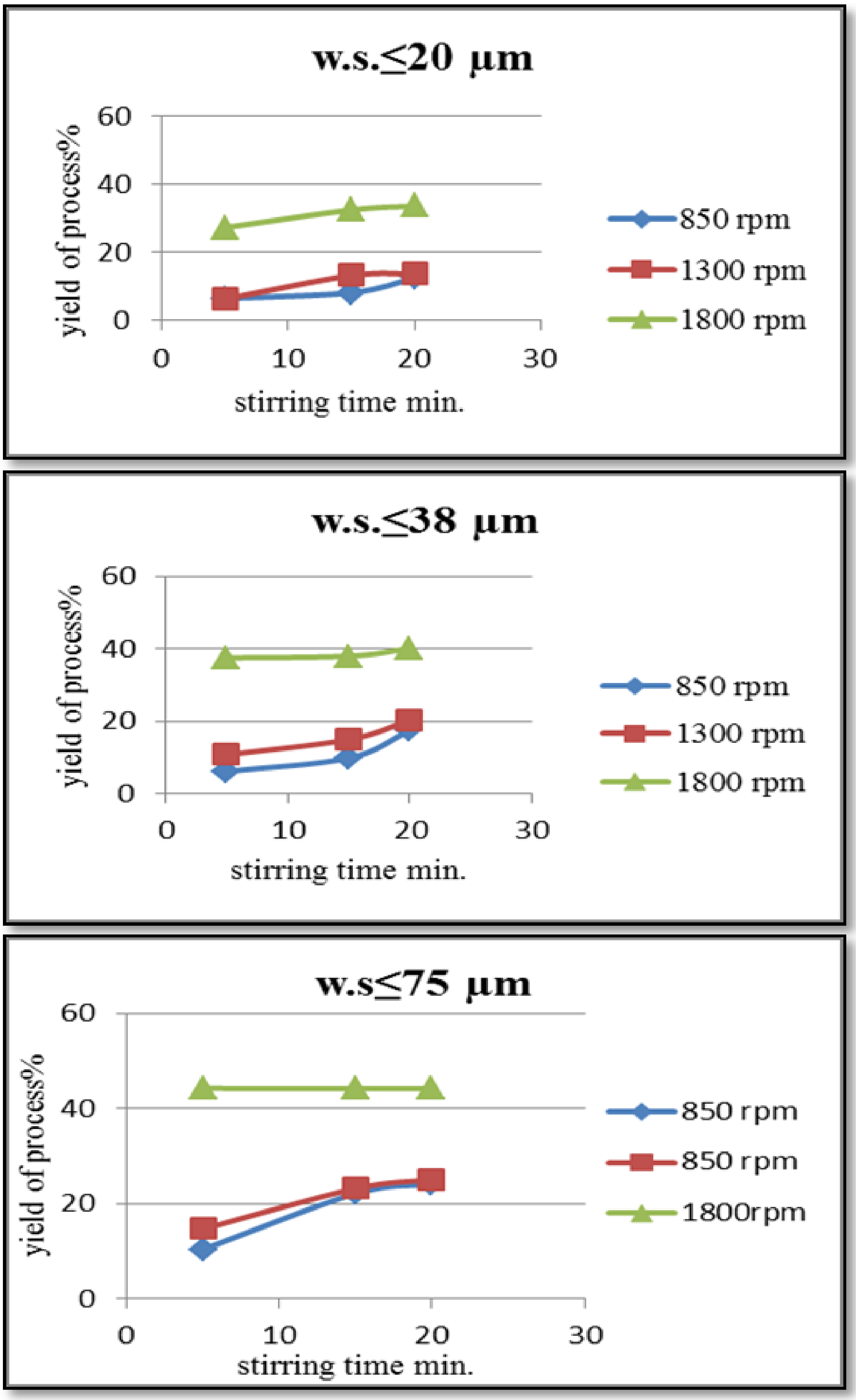

Figure2. The relationship between yield of process and stirring time at different mesh size.

3.3 FE-SEM image Result:

The FE-SEM images of the prepared attapulgite are shown in figure 3. Those clarify the impact of sieve size on attapulgite rods dispersion. Figure 3(a-c) show the effect of mesh size 75µm (mesh no.200) shown in figure 3(a-c), can be seen that the attapulgite rods exist as a crystal bundle, these crystal bundles were naturally grouped to form aggregates [15]. also, there was not clear dispersion for attapulgite rods or its bundles when sieved with 75µm mesh size. As a result of wet sieving with mesh size 38 µm (mesh no.400); attapulgite rods exist in two forms; first attapulgite rod were existent as a crystal bundles with high alignment level as shown in figure4e. Second attapulgite rods were dispersed and re grouped as small aggregates due to the physical bounding force (electrostatic, hydrogen bonding and van der Waals) as shown in figure 3(f and g).

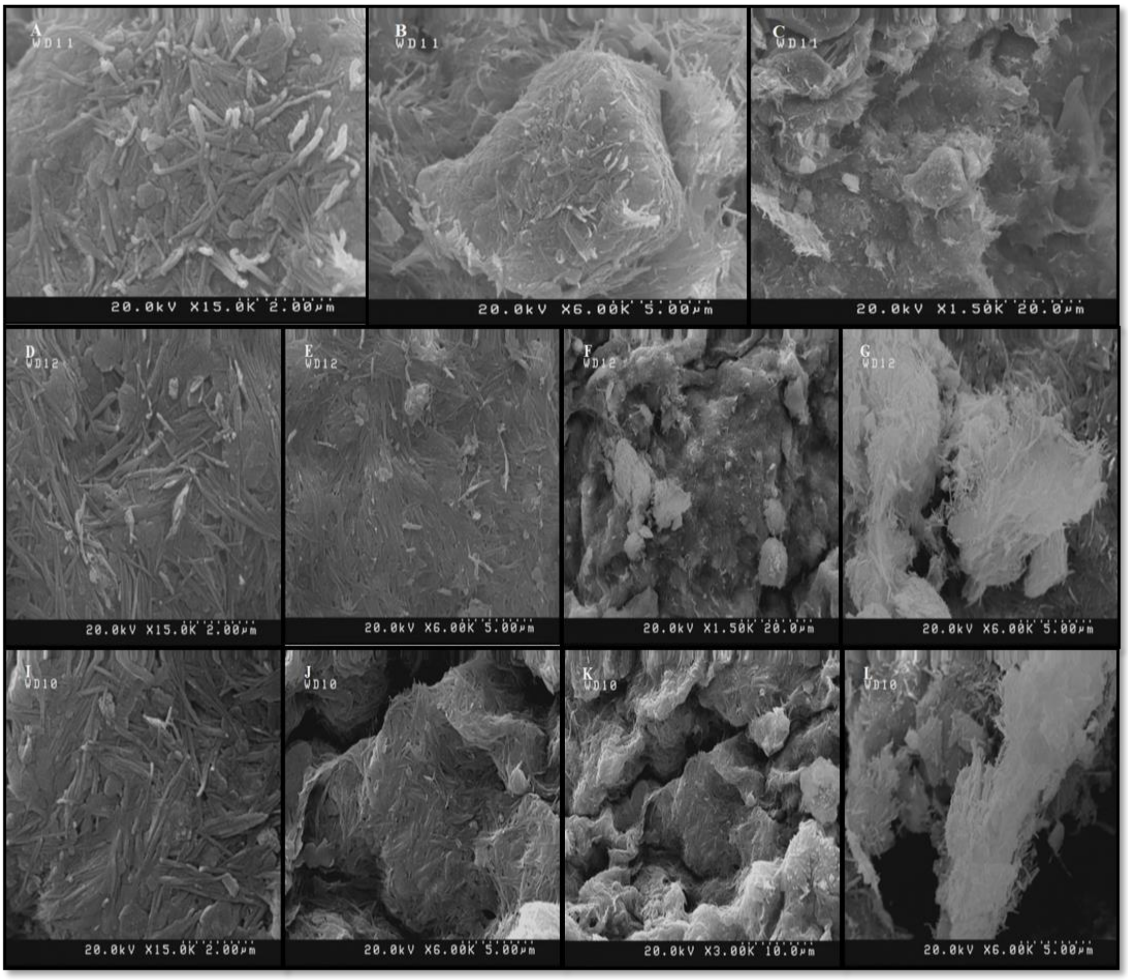

Figure 3. FE-SEM images (a-c) mesh size 75µm. (d-g) mesh size38µm. (i-l) mesh size20 µm.

Comparing with attapulgite that sieved with 38 µm mesh size; sieving process with mesh size 20 µm (mesh no.=635) was clearly activate dispersion of attapulgite crystal bundles, where attapulgite aggregates existed in smaller size and wide distribution as shown in figure 3(I –L). However, the alignment of attapulgite crystal bundles was disappeared as a result of increasing attapulgite dispersion level.

Generally, the main reason for attapulgite dispersion increasing in wet sieving process is the clay friability. Where; friability is the particle's propensity to divide. Stirring and sieving may alteration the particles size distribution for friable material [16].

In this work, when attapulgite suspension is wet sieved; attapulgite aggregate will be divided to smaller size under the action of sieving process. Theoretically, mesh number will represent aggregates number of attapulgite clay within suspension. So there is an inverse relationship between the dispersion of attapulgite and the mesh size. By other way, with increasing mesh number (decreasing mesh size); attapulgite dispersion will increase, while the size of attapulgite aggregates is decrease.

Later, the resulted smaller aggregates from sieving process will re-aggregate under the acting of physical bounding force (electrostatic, hydrogen bonding and van der Waals). The size of those new aggregates was proportion with mesh size. Thus, it became clear why attapulgite dispersion increased with the mesh number in wet sieving process.

3.4 Rheological properties results

In this work, the quality estimating of attapulgite clay as drilling fluid was implemented depending on the value of apparent viscosity and yield of clay for sieved attapulgite clay as shown in figure 4 and table 2.

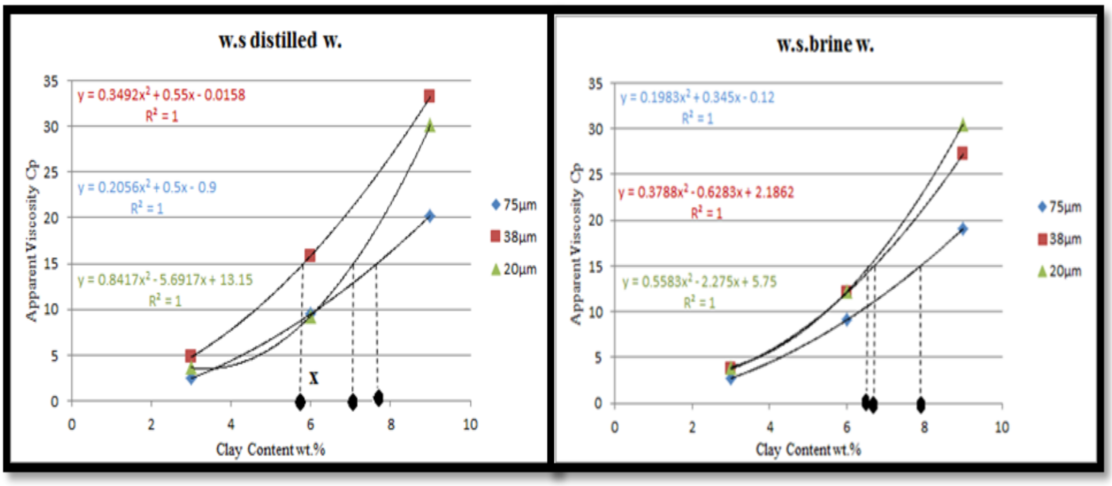

Figure 4. The relationship between apparent viscosity and clay content.

Apparent viscosity of attapulgite base drilling fluid had affected by many factors such as aspect ratio (l/d), clay concentration, adsorption ion, PH and dispersion level, at the same time, some of these factors were affected with other different factors [3].

Table2. Rheological properties for purified attapulgite clay.

	75µm		38µm		20µm	
	D.W	S.W	D.W	S.W	D.W	S.W
PV	1.5	1.2	3.2	2.03	1.06	2.12
YP	8.17	8.02	12.87	10.25	8.41	10.29
10 S gel	8.05	7.2	13.2	7.66	12.83	9.45
10 min gel	8.03	7.9	13.3	8.66	15.93	8.97
A.V	9.5	9.05	15.83	12.06	9.3	12.2
Stability	61.5	-	69.5	-	62.5	-
Yield of clay	71.07	68.91	94.21	81.71	77.61	83.26
Clay %	7.68	7.91	5.86	6.72	7.06	6.6
PH	7	7	7	7	7	7

The appearance of an effect of these factors on apparent viscosity is associated with the applied laboratory variables (combined or individually). In this work, the impact of sieving process was clear on dispersion of attapulgite crystal bundles, hence, on the apparent viscosity. Sieved attapulgite clay with 38 µm mesh size had higher apparent viscosity and higher yield of clay, this could be due to the high alignment level and the small size of attapulgite re-aggregates which mean increasing in active surface area. While sieving with 20 µm mesh size leads to increase dispersion of attapulgite crystal bundles (aggregates smaller size and wide distribution). Moreover, alignment missing for crystal bundles causing reduction in apparent viscosity. Therefore, the sieved attapulgite clay with20µm mesh size comes at second grade.

However, there is a physical rule explains the relation between dispersion and rheological properties for the above results which is (if particles surface area to mass ratio increased significantly, the surface properties tend to take control in system leading to aggregate particles, Consequently, changes were made to the rheological properties) [17].

Sieving at 75µm mesh size is the agreed sieve size in standard drilling fluid preparation process no other size, therefore it was considered the initial attapulgite clay for wet sieving process. Anyway, sieving at 75µm mesh size had no significant influence on attapulgite dispersion; for that reason, it had the lowest apparent viscosity and lowest yield of clay. Which represent the initial value for the origin sieved attapulgite clay.

According to the result of rheological properties and yield of process, the best results were at milling speed 1800 rpm for20 min. and sieving with mesh size 38 µm as shown in Figure 5.

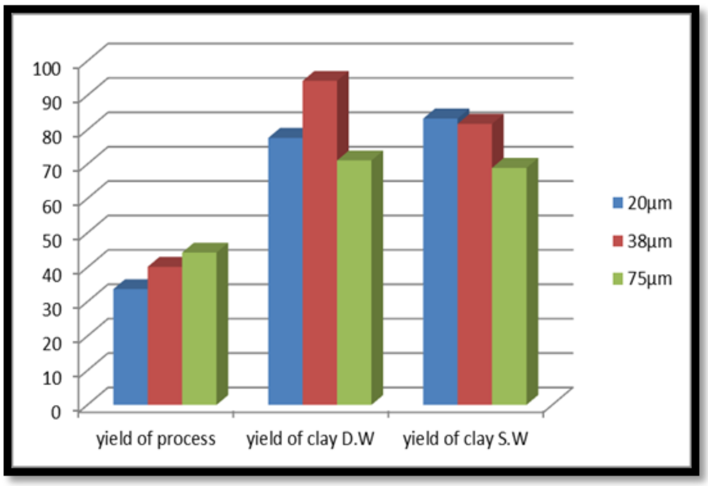

Figure 5. The qualitative and quantitative properties of produced attapulgite clay at best milling conditions.

4.4 Sedimentation (Stability) result

The sedimentation behavior for sieved attapulgite clay at all tested sizes were reach stable sedimentation volume after 60 hours as shown in Figure 6. Also; sedimentation curves for sieved attapulgite with 75µm mesh size and 20 µm mesh were the same. This could be explained by the converge in apparent viscosity values for the two sizes (75 and 20 µm). Sieved attapulgite with 38µm mesh size has the larger sedimentation volume; this could be due to higher apparent viscosity value as explained above.

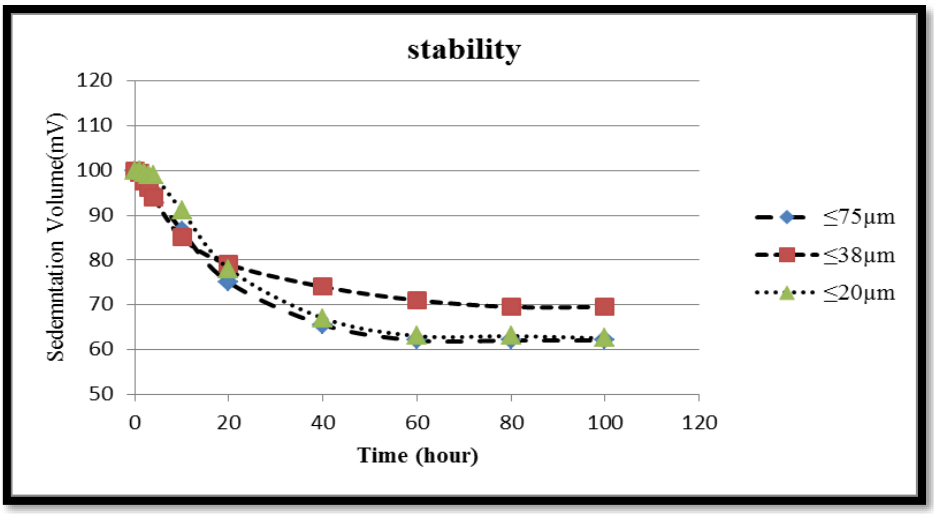

Figure 6. sedimentation behavior of wet sieved attapulgite at different mesh sizes.

- Conclusion

Wet sieving is an active clay separation process. Wet sieving conditions (stirring speed, stirring time and mesh size) were active tool in controlling the properties of final product. At the same time, quality and quantity of prepared attapulgite clay were adopted in fixing conditions of wet sieving process to prepare attapulgite clay for oil wells drilling fluid. From this work many outcomes can be concluded:

- Stirring time and stirring speed had a positive affect to increase liberation of attapulgite. Thus, yield of process was seriously increased.
- Yield of process had direct proportional relationship with mesh size.
- Higher yield of process obtained at 1800 rpm stirring speed, 20 minutes stirring time and at mesh size 75 µm.
- Impact of mesh size on attapulgite dispersion was very clear especially at mesh size 38 and 20µm.
- According to rheological properties, there was critical mesh size; 38 µm gives higher apparent viscosity and higher yield of clay.
- Finally, figure 7 shows the recommended process layout to prepare Iraqi attapulgite clay (Bahar AL Najaf region) by wet sieving process to be suitable for oil wells drilling fluid.

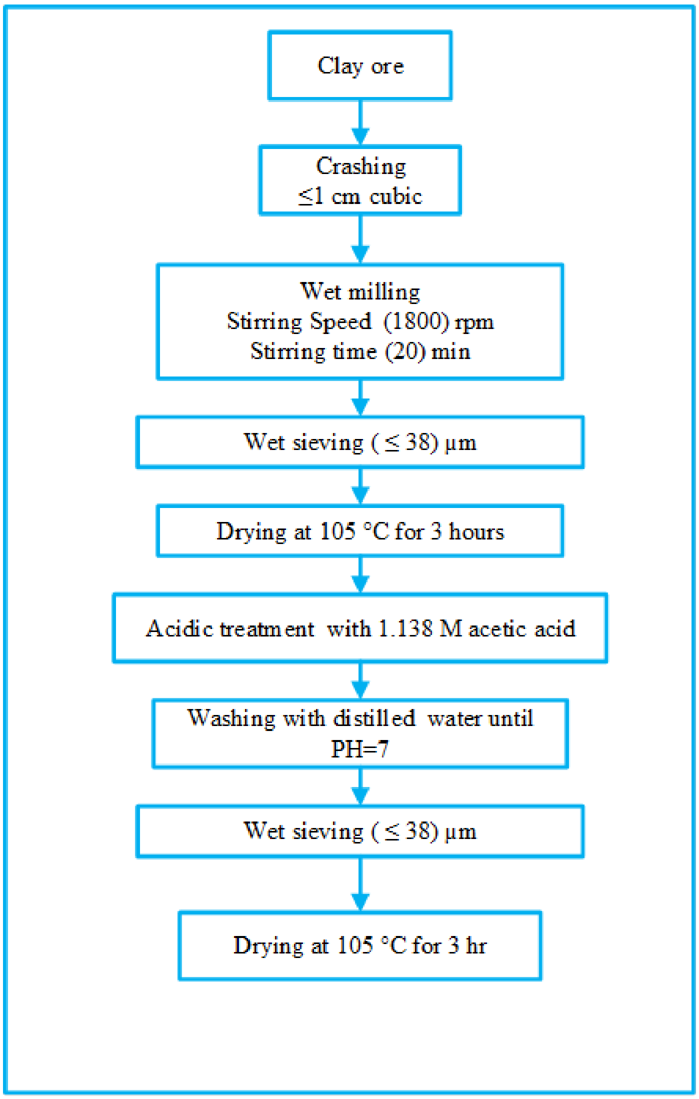

Figure 7. Recommended wet sieving process layout.

CONFLICT OF INTERESTS.

- There are no conflicts of interest.

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تحضير طين الأتبلغايت العراقي بأستخدام عملية الغربلة الرطبة ليكون مناسباً لسوائل حفر آبار النفط

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الخلاصة:

الغربلة الرطبة هي احدى عمليات الفصل الفعالة للمعادن الطينية، في هذا العمل، استخدمت عملية الغربلة الرطبة لفصل طين الأتبلغايت عن الكوارتز الموجود في التربة العراقية ليكون مناسب للأستخدام في سوائل حفر الآبار النفطية. تم دراسة تأثير الظروف التشغيلية لعملية الغربلة الرطبة (سرعة الخلط، زمن الخلط وقياس فتحة الغربيل) على كلا من الخواص الكمية والنوعية لطين الأتبلغايت. في عملية الطحن الرطب، استخدمت سرع خلط مختلفة (٨٥٠ ،١٣٠٠، ١٨٠٠ (دورة/دقيقة في ازمان خلط مختلفة (٥، ١٥، ٢٠) دقيقة. بلأضافة الى ذلك، ثلاث غرابيل بقياسات مختلفة (٣٨،٢٠، ٧٥) مايكرومتر استخدمت في عملية الغربلة الرطبة. حيود الأشعة السينية ومساحة الأنبعاث- المجهر الألكتروني الماسح استخدمت لغرض تشخيص الخواص المعدنية والسطحية لطين الأتبلغايت. الخواص الريولوجية وانتاجية طين الأتبلغايت تم قياسها في الماء العذب والماء المالح بأستخدام مقياس اللزوجة ماركة (Ofite). انتاجية عملية الغربلة الرطبة قيست بأخذ الفرق بوزن الطين قبل عملية الغربلة وبعد عملية التنقية. اظهر النتائج بأن سرعة الخلط وزمن الخلط لهما تأثير كبير في زيادة انتاجية العملية، بحيث اعلى انتاجية للعملية تم الحصول عليها عند سرعة خلط ١٨٠٠ دورة/دقيقة لمدة ٢٠ دقيقة بأستخدام غربيل بحجم فتحة ٧٥ مايكرومتر. كذلك اظهرت النتائج ان لحجم فتحة الغربيل تناسب طردي مع انتاجية العملية، بينما لها تناسب عكسي مع مستو تشتيت طين الأتبلغايت. على اي حال، أفضل قيم لزوجة ظاهرية كانت (١٥,٨٣و١٢,٠٦سانتي بويز) في الماء العذب والماء المالح على التوالي. اعلى انتاجية للطين كانت (٩٤,٢١و ٨١,٧١ برميل/طن) في الماء العذب والماء المالح على التوالي تم الحصول عليها بأستخدا غربيل ذو حجم فتحة٣٨مايكرومتر. اخير الظروف التشغيلية لعملية الغربلة الرطبة هي سرعة خلط ١٨٠٠ دورة/دقيقة وزمن خلط ٢٠ دقيقة وغبيل ذو حجم فتحة ٣٨ مايكرومتر.

الكلمات الدالة: – طين الاتبلغات العراقي، طريقة الغربلة الرطبة، الخواص الريولوجية، سرعة الخلط، حجم فتحة الغربال.