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Beneficiation of Low Grade Calcium Bentonite Claystone

Abstract- *The beneficiation of Ca-bentonite claystone has been studied with dispersion sedimentation technique using polyionic salts as dispersant.The claystone is located in the Western Desert of Iraq. It is of a low grade, associated with different amount of clay and non-clay mineral impurities. Calcite ($CaCO_3$) constitutes the major proportion of these impurities. Various parameters; solid concentration, dispersant amount (e.g. sodium tripolyphosphate, and tetrasodium pyrophosphate), conditioning time, and centrifugal sedimentation speed and time on the efficiency of the beneficiation process were investigated and followed through the measurement of CaO% and cation exchange capacity (CEC) values of the upgraded claystone concentrate. Centrifugal sedimentation were tested to separate the impurities from the clay suspension. Design experiments by Taguchi method, orthogonal array L16, was used for optimizing the different process parameters of the beneficiation process. Experiments were conducted at different solid concentrations (1, 3, 5, 7) wt.%, dispersion agent amount (0.2, 0.4, 0.6, 0.8) wt.%, conditioning time (5, 10, 15, 20) min, centrifugal sedimentation speed (500, 600, 700, 800) rpm, and centrifugal time (5, 10, 15, 20) min. The optimum beneficiation conditions obtained from the experimental work are, 7 wt.% solid concentration, 0.8 wt.% of dispersant, 15 min conditioning time attachment of the dispersant agent with the bentonite slurry, 800 rpm centrifugal speed, for 10 min time. Under studied condition, tetrasodium pyrophosphate showed better output t for achieving good beneficiation of bentonite clay from dilute crude slurry.*

Keywords-*beneficiation, cation exchange capacity (CEC), centrifugal sedimentation, dispersion, montmorillonite.*

1. Introduction

Montmorillonite occupies a prominent position among industrial minerals. It is the main constituent of bentonite and the detrimental factor of its properties. Regardless of origin or occurrence, montmorillonite and bentonite are two names of exactly the same mineral [1]. The electrical neutrality is attained by other cations (external to the lattice) that reside in the inter-laminar region, between the lattice layers. These cations (usually Na^+ or Ca^{2+}) are relatively easily exchangeable, giving montmorillonite high cation exchange capacity phenomenon [1-4]. The type of cation that fulfils the charge-balancing role has marked impact on the performance of the bentonite in terms of its capacity for swelling, thixotropic, and adsorption [2,5]. In industry, bentonite is generally either sodium (Na^+) or calcium (Ca^{2+}) type. Sodium bentonite, where the inter-laminar region is occupied mainly by sodium ions dominant exchangeable ion, has very high swelling capacities and thixotropic properties when added to water. According, it has special application in allied drilling muds and other uses requiring thixotropic suspension. On the other hand, Ca-bentonite has no value for such application. It has a very little swelling ability; flocculation and settling are much more rapid than for sodium bentonite [6-8]. In Iraq, particularly in the Western Desert, bentonite was first reported by Al-Bassam, and Al-Saadi [9]. Years later, the exploratory works resulted for localizing two calcium bentonite claystone deposits [10-11]. The deposit, located in Wadi Bashira as shown in Figure 1, was considered the major deposit explored in the Western Desert. It represents the lower part of the Digma Formation of Late Cretaceous age and of marine sedimentary origin. It is of low grade with montmorillonite content associated with clay and non-clay impurities including attapulgite, calcite, quartz, apatite, gypsum, and halite. Calcite, however, represents the major impurity, which is around 15 wt.% of the deposit [11]. The bentonite claystone deposit, showed a poor response to sodium activation and failed for using in drilling mud when tested under

(API specification) [12]. Generally, the major problems facing the utilization of Wadi Bashira claystone deposit are its low concentration of bentonite (about 68%), high level of impurities, and inconsistent composition. Therefore, it has to be processed and upgraded before utilization. The potential method for this purpose is the wet beneficiation method [2]. It has been demonstrated that by others methods Ca-bentonite slurry to a hydrocyclone classifier and treating the over flow with dilute HCl, led to effective removal of calcite impurities. Similar results were achieved when slurry of Ca-bentonite claystone was treated with dilute HCl to remove calcite impurities [14]. Furthermore, it was shown that dispersion and sedimentation processes with sodium phosphate as dispersant was very effective in beneficiating China Ca-bentonite claystone [15]. The reject from this process was mainly quartz sand. It was also reported that dispersion sedimentation technique with tetrasodium pyrophosphate (TSPP) was very effective in separating carbonate impurities from Iraqi bentonite claystone deposit, and the reject was mainly calcite [16]. The objective of the present work is, to study the beneficiation potential of Wadi Bashira bentonite claystone, using dispersion sedimentation method [17], and to establish the best condition of the process parameters e.g. dispersant addition, slurry stirring time, solid concentration and centrifugal separation (speed and time).
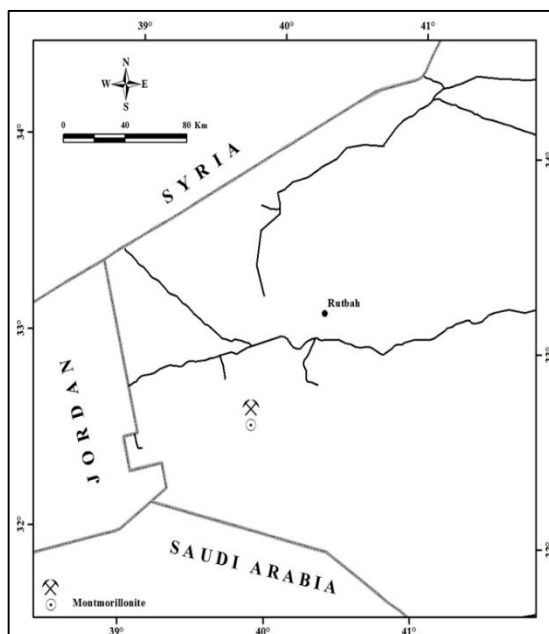
Figure 1: Location map of bentonite clay deposit of Wadi Bashira in the western Desert of Iraq [17].

2. Experimental Work

I. Sample Preparation

Ca-bentonite sample from Wadi Bashira, in Western Desert of Iraq, was received from the Geological Survey of Iraq for the beneficiation tests. The claystone sample, as received, was successively crushed to pass minus 1 mm sieve in laboratory jaw crusher. Identical samples of 0.5 Kg each were isolated for further treatment using Jons Riffle sampler. Furthermore, a representative specimen was withdrawn for chemical, XRD and cation exchange capacity (CEC) analysis. The CEC was determined by the methylene blue adsorption method, and was used to evaluate the bentonite purity of both. The procedure used was European Standard [3]. Sample from the prepared claystone was drawn for chemical and mineralogical analysis.

II. Upgrading of Montmorillonite Claystone

The employed beneficiation method comprised the following steps:

1. Preparing a slurry of the crushed montmorillonite claystone containing 7 wt.% solid and then degritting the slurry on 75 μm to remove the oversize +75 μm fraction.

2- Adding dispersant to the degritted slurry to disperse the clay in water. The dispersing agents used in this work were; Tetrasodium pyrophosphate (TSPP) $Na_4P_2O_7.10H_2O$, and Sodium tripolyphosphate (STPP) - $Na_5P_3O_{10}$.

3- Separating the dispersed clay from undispersed (settled) material by decantation after centrifugal sedimentation.

4- For dispersion addition amount, stirring slurry with dispersants for condition mixing time (5, 10, 15, and 20 min) and then the formation (clay suspension) was separated by decantation.

V. For centrifugal sedimentation, various speeds i.e. 500, 600, 700 and 800 rpm and time intervals of 5, 10, 15 and 20 min were applied.

5- Dewatering the decanted dispersed clay was achieved at high speed centrifugation of 3000 rpm.

6- Drying of the separated clay was performed at 100 ℃ ± 5 ℃ and then analyzing the dried clay for CaO and CEC value.

III. Taguchi Experimental Design

In order to determine the optimum beneficiation conditions during beneficiation experiments, design of experiments by Taguchi method was used. The studied parameters are dispersant amount, conditioning time (mixing time of dispersant and clay slurry), solid concentration (amount of bentonite clay in the slurry), centrifugal time intervals and each of the previous

parameters has four levels. Minitab program is chosen to select and create the proper Taguchi orthogonal arrays. There parameters and their levels however were designed statistically, as shown in Table 1. Taguchi orthogonal arrays L16 (4^5) which is shown in Table 2, is selected according to the number of parameters and their level.

Table 1: Processing factors and their levels of beneficiation Ca-bentonite clay.

Processing Parameters	Levels of parameters			
	Level 1	Level 2	Level 3	Level 4
(A) Solid concentration, wt.%	1	3	5	7
(B) Dispersion agent, wt.%	0.2	0.4	0.6	0.8
(C) conditioning time, min	5	10	15	20
(D) centrifugal speed, rpm	500	600	700	800
(E) centrifugal time, min	5	10	15	20

Table 2: Taguchi orthogonal array L16 (4^5) for beneficiation parameters.

Exp. No	Solid concentration	Dispersion agent Wt %	Conditioning time	Centrifugal speed	Centrifugal time min
1	1	0.2	5	500	5
2	1	0.4	10	600	10
3	1	0.6	15	700	15
4	1	0.8	20	800	20
5	3	0.2	10	700	20
6	3	0.4	5	800	15
7	3	0.6	20	500	10
8	3	0.8	15	600	5
9	5	0.2	15	800	10
10	5	0.4	20	700	5
11	5	0.6	5	600	20
12	5	0.8	10	500	15
13	5	0.2	20	600	15
14	5	0.4	15	500	20
15	5	0.6	5	700	5
16	5	0.8	10	800	10

3. Results and Discussion

1. Mineralogical and Chemical Composition

According to the mineralogical analysis of the raw claystone sample determined by X- ray diffraction depicted in Figure 2, the predominant identified mineral phases: calcite, quartz, attapulgite and montmorillonite. Minor amounts of gypsum and feldspar were also observed. Table 3 presents the chemical composition of the raw claystone sample. It is show that the chemical composition of attapulgite and montmorillonite in the Digma

Formation is sufficiently close and these clays can hardly be identified from each other by chemical analysis. However, according to the data shown in Table 3, the raw claystone sample displays a high content of impurities, especially calcite, which is reflected by the high amount of CaO in the sample. The presence of the carbonate as well as some gypsum reflects the high loss on ignition (L.O.I) value of the raw claystone.

Table 3: Chemical composition of the investigated Ca- Bentonite claystone raw sample.

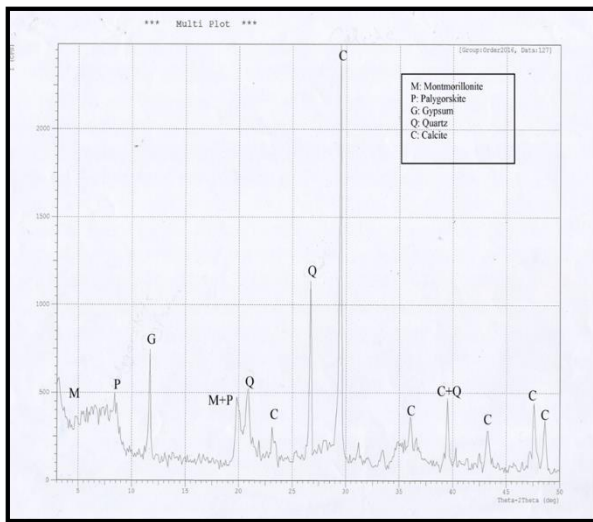| SiO$_2$ %	Fe$_2$O$_3$	Al$_2$O$_3$	CaO %	MgO %	SO$_3$ %	Na$_2$O %	K$_2$O %	Cl	L.O.I
37.84	5.17	11.60	17.74	3.45	4.60	0.94	0.29	0.75	17.74

Figure 2: XRD pattern of the raw claystone sample

II. Results Analysis and Identifying the Optimum Conditions

After finishing all beneficiation experiments, results are entered in the work sheet of Minitab program for analysis. Tables 4 and 5 show Taguchi orthogonal arrays with its results, means of CaO% and cation exchange capacity (CEC) for (STPP) and (TSPP) as dispersants in the beneficiation process, respectively. The optimum condition for each factor is determined by comparison between mean of beneficiation efficiencies figures.

Table 4: Results analysis of (CaO% and CEC) obtained from using (STPP) as dispersant.

Exp. No.	Solid concentration Wt.%	Dispersion agent Wt.%	Conditioning time min	Centrifugal speed rpm	Centrifugal time min	Mean $\sum$ CaO	Mean $\sum$ CEC
1	1	0.2	5	500	5	12.9225	58.8275
2	1	0.4	10	600	10	10.7725	67.6650
3	1	0.6	15	700	15	9.6100	73.7125
4	1	0.8	20	800	20	6.9250	84.4425
5	3	0.2	10	700	20	11.3025	68.8925
6	3	0.4	5	800	15	8.105	75.2000
7	3	0.6	20	500	10	9.7075	55.0475
8	3	0.8	15	600	5	6.7300	87.5425
9	5	0.2	15	800	10	9.9525	71.2650
10	5	0.4	20	700	5	10.4650	70.4075
11	5	0.6	5	600	20	8.7275	75.7100
12	5	0.8	10	500	15	5.9200	87.5400
13	5	0.2	20	600	15	11.0975	71.8500
14	5	0.4	15	500	20	10.4650	76.2250
15	5	0.6	5	700	5	6.4725	87.6825
16	5	0.8	10	800	10	5.1450	90.6975

Table 5: Results analysis of (CaO% and CEC) obtained from using (TSPP) as dispersant

Exp. No.	Solid concentration Wt.%	Dispersion agent Wt.%	Conditioning time min	Centrifugal speed rpm	Centrifugal time min	Mean $\sum$ CaO	Mean $\sum$ CEC
1	1	0.2	5	500	5	15.5675	59.4550
2	1	0.4	10	600	10	10.6375	75.2225
3	1	0.6	15	700	15	9.5800	76.5400
4	1	0.8	20	800	20	5.7925	82.7925
5	3	0.2	10	700	20	12.5400	65.5950
6	3	0.4	5	800	15	9.3825	75.1000
7	3	0.6	20	500	10	10.437	81.2600
8	3	0.8	15	600	5	4.3150	82.8175
9	5	0.2	15	800	10	10.5550	75.2825
10	5	0.4	20	700	5	11.2100	68.2825
11	5	0.6	5	600	20	9.8700	79.6575
12	5	0.8	10	500	15	4.1025	89.1525
13	5	0.2	20	600	15	11.9300	68.7300
14	5	0.4	15	500	20	7.7675	81.2450
15	5	0.6	5	700	5	6.6075	85.9350
16	5	0.8	10	800	10	2.1950	98.3375

III. Effect of Solid Concentration

To investigate the influence of solid concentration on beneficiation process, beneficiation experiments are done in levels of (1 to 7) wt.%. The effect of various raw bentonite clay concentration (bentonite claystone ore concentration) on CaO% of the upgrading clay for (STPP) and (TSPP) as dispersants are illustrated in Figure 3 and 5, respectively. It can be observed from these figures, that at 1 wt.% of the solid concentration the CaO% of the beneficiated bentonite clay was lowered for 17.74% CaO to about 10.1% CaO for (STPP) and to 10.5% of CaO for (TSPP). This obviously led to increase in the CEC value in Figures 4 and 6 from 57 meq/100 gm to about 71.162 meq/100 gm values for (STPP) and 73.5 meq/100 gm of the yielded bentonite concentrate. However, the mean lower of CaO (8.3 and 7.1%) and higher to about (81.6 and 83.6) meq/100 gm CEC value for (STPP and TSPP) were obtained at 7% solid concentration. That the CaO value of the yielded clay concentrate gradually increase as the solid concentration increase from 1 wt.% to 7 wt.%. It can be deduced that good upgrading of bentonite can be achieved at solid concentration 7% by weight.

IV. Effect of Dispersant Addition

The effect of the dosage of sodium tripolyphosphate (STPP) and tetrasodium pyrophosphate (TSPP) to the raw bentonite clay slurry to enhance its dispersion and thereafter on CaO% of the beneficiated bentonite is presented in Figures 3 and 5. The results indicate that CaO percentage of (11.3% and 12.6%) were obtained with the addition of 0.2% (STPP and TSPP). This value was sharply reduced with the dispersant addition from (0.2- 0.8) wt.%. At added 0.8 wt.%, the lower CaO percentage were achieved for (6.18% and 4.1%). The decrease in CaO% value, however, was compensated by the increase of the CEC value of the bentonite concentrate as shown in Figures 4 and 6 which were about (87.6 and 88.3) meq/100 gm at the addition of 0.8 wt.% (STPP and TSPP) dispersants agent. Therefore, this can be considered as optimum value. However, the visual observation of the mode of sedimentation and settling rate of the impurities indicate that the clay slurry become more fluid with the increase of the dispersant addition, with the range tested.
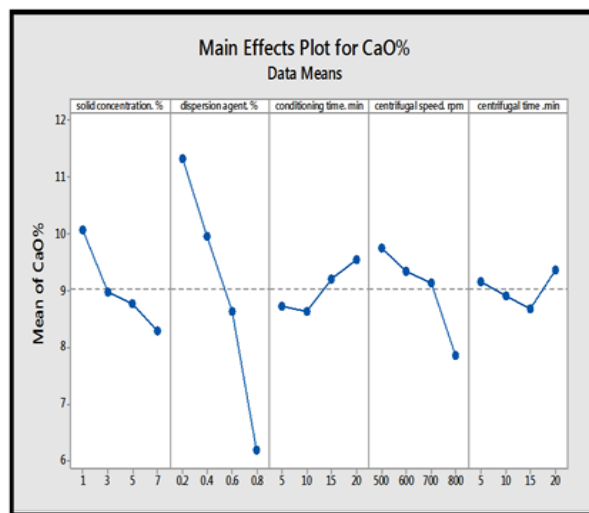

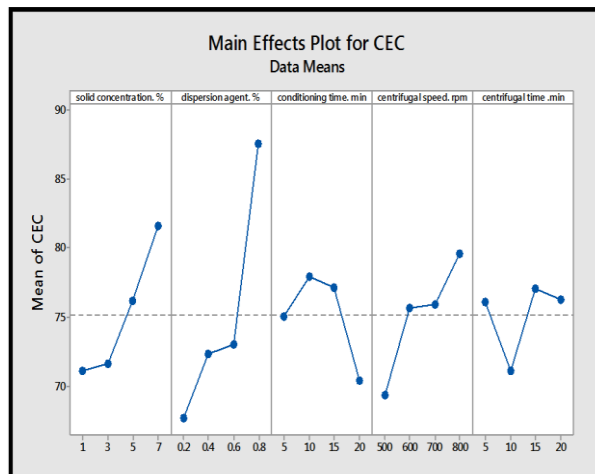**Figure 3: The effect of beneficiation factors on CaO% content using (STPP) as dispersant**

Figure 4: The effect of beneficiation factors on CEC value using (STPP) as dispersant.
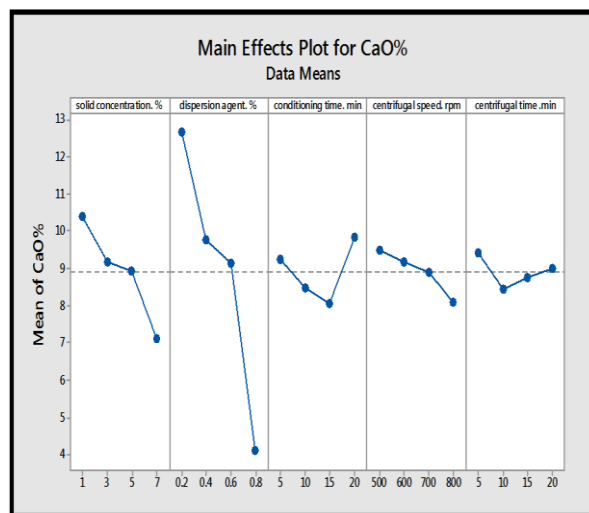

Figure 5: The effect of beneficiation factors on CaO% content using (TSPP) as dispersant.
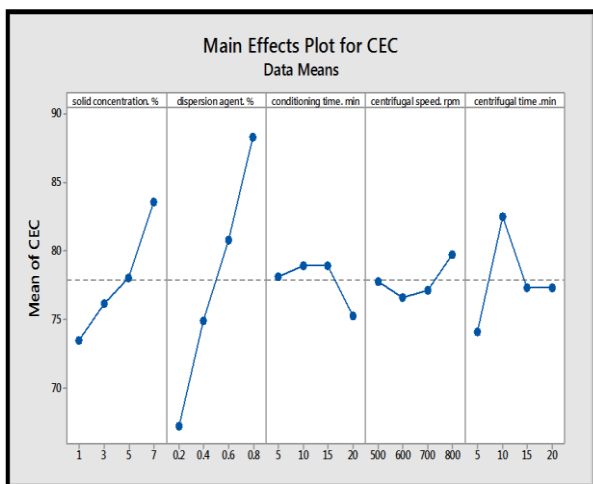
Figure6. The effect of beneficiation factors on CEC value using (TSPP) as dispersant.

V. Effect of Conditioning Time

To study the influence of conditioning time between the dispersing reagent and clay particles is very beneficial for wetting clay particles and allow the dispersant ion to chemisorbed at the edge and surface of the clay particles. Thereupon increases the electrical double layer repulsion between the clay mineral (montmorillonite) particle surfaces in the slurry and induces its suspension [6,15]. The influence of conditioning time on CaO% of the upgrading bentonite produced are illustrated in Figures 3 and 5, respectively. The results indicate that at 10 min conditioning time, the CaO content of the yielded bentonite was reduced to about 8.6% for (STPP) and at 15 min, the CaO% reduced to 8.1% for (TSPP), and subsequently its CEC value in figures 4 and 6 accompanied by sharp increasing in in its CEC occur. The CEC value obtained was of about (77.9) meq/100 gm for (STPP) and to (78.9) meq/100 gm for (TSPP). With further increase in the conditioning, time no significant effect occured neither on the CaO% nor on the CEC value of the beneficiated claystone. Therefore, conditioning time of (10) min when add (STPP) and (15) min for (TSPP) was considered as optimum time interval needed for the clay dispersing step.

VI. Effect of Centrifugal Sedimentation

After the dispersion of the clay in water and to facility the sedimentation (separation) of non-clay impurities from the clay suspension, centrifugal sedimentation was tested. Therefore the next step in the investigation was to optimize the time and speed of centrifugation that can yield a high-grade bentonite clay concentrate. The tests were conducted using various speeds (500, 600, 700, and 800 rpm) and various centrifugal times (5, 10,

15 and 20 min). The influence of these parameters on the CaO% and CEC value on the beneficiation of Ca-bentonite claystone are illustrated in Figures 3 and 5 for CaO% and Figures 4 and 6 for CEC, respectively. The results indicate that (9.8% and 9.2%) CaO for (STPP and TSPP) with using lower speed 500 rpm for 5 min showed very little sediment of impurities that quickly disbanded i.e. unstable and cannot be separated. Increasing the speed to 800 rpm observed that access CaO to about 7.9% and to about 8.7% for 15 min when using (STPP) as dispersant and to about (8.1% and 8.2%) for 10 min when using (TSPP) to beneficiation process. This was supported by the increase of the CEC of the bentonite concentrate which was increased to about (79.648) meq/100 gm and (77.076) meq/100 gm with 15 min for (STPP) and to about (79.8 and 82.5) meq/100 gm with 10 min for (TSPP). Therefore, this speed (800 rpm) and 10 min were considered as optimum for the centrifugal sedimentation operation when using (TSPP) as dispersant more than (STPP).

VII. Optimum Beneficiation Conditions

In order to determine the optimum conditions for the beneficiation process, reflected or represented by CaO% and CEC value found through experiments as indicated in Tables 6 and 7. Four experiments were conducted using (STPP) and (TSPP) as dispersants under optimum conditions for the purpose of confirmation. The optimum beneficiation conditions obtained from the experimental work are, 7 wt.% solid concentration, 0.8 wt.% of dispersant, 15 min conditioning time attachment of the dispersant agent with the bentonite slurry, 800 rpm centrifugal speed, for 10 min time when using (STPP) as dispersant in the beneficiation process, while the optimum beneficiation conditions obtained using (TSPP) as dispersant are, 7 wt.% solid concentration, 0.8 wt.% of dispersant, 10 min conditioning time attachment of the dispersant agent with the bentonite slurry, 800 rpm centrifugal speed, for 15 min time.

Table 6: The optimum beneficiation experiment results for (STPP) dispersant

No. of parameters		CaO %	CEC meq/100 g
(A) Solid concentration,	wt.%	5.11	90.65
(B) Dispersion agent,	wt.%	5.10	91.57
(C) conditioning time,	min	5.09	90.71
(D) centrifugal speed,	rpm	5.04	90.58
Mean		5.01	90.87

Table 7: The optimum beneficiation experiment results for (TSPP) dispersant

No. of parameters		CaO %	CEC meq/100 g
(A) Solid concentration,	wt.%	2.13	98.21
(B) Dispersion agent,	wt.%	2.25	97.40
(C) conditioning time,	min	2.16	98.29
(D) centrifugal speed,	rpm	2.20	98.60
Mean		2.185	97.875

VIII. Analysis of Variance (ANOVA)

The effective percentage for each factor on the CaO% and CEC value for beneficiation process are calculated by Minitab program. The ANOVA calculation for CaO% and CEC value are illustrated in tables 8 and 9 when using (STPP) as dispersant, respectively. And tables 10 and 11 when using (TSPP) as dispersant.According to ANOVA results, it is observed that the percentage of dispersion agent and solid concentration have significant effects on the reduced amount of the

CaO% and increased the cation exchange capacity (CEC) more than the centrifugal time, centrifugal speed, and conditioning time when using (STPP) and (TSPP) as dispersants. The chemical composition results of the upgraded montmorillonite claystone were obtained at the optimum conditions (7 wt.% solid, 0.8 wt.% TSPP, 15 min conditioning time, 800-rpm centrifugal speed, 10 min centrifugal time). It was found when using (TSPP) as dispersant in upgrading Ca-bentonite is much better than when using (STPP), accordingly, the upgraded bentonite claystone can be considered as a high-grade product. The purity was also reflected by the values of CEC, which were found to be 98.20 meq/100 gm respectively. Figure 7 shows the X-ray diffraction patterns of the claystone sample after being beneficiated by the dispersion and sedimentation processes. The chemical composition is shown in Table 12 which show low amount of CaO to be 2.15%.

Table 8: ANOVA results of L16 for (CaO%) using (STPP) as dispersant.

Source of variation	Degree of freedom	Sum of squares	variance	P contribution
Solid concentration	3	6.679	2.226	8.863
Dispersion	3	57.483	19.161	76.269
Condition time	3	2.231	0,744	2.960
Centrifugal speed	3	7.939	2.646	10.534
Centrifugal time	3	1.031	0,344	1.368
Error	0			
Total	15	75.3642		100

Table 9: ANOVA results of L16 for (CEC) value using (STPP) as dispersant.

Source of variation	Degree of freedom	Sum of squares	variance	P contribution
Solid concentration	3	283.83	86.643	17.629
Dispersion	3	885.74	368.978	55.016
Condition time	3	136.69	8.134	8.490
Centrifugal speed	3	216.29	14.956	13.435
Centrifugal time	3	87.40	3.687	5.429
Error	0			
Total	15	1609.95		100

Table 10: ANOVA results of L16 for (CaO%) using (TSPP) as dispersant.

Source of variation	Degree of freedom	Sum of squares	variance	P contribution
Solid concentration	3	217..32	72.440	15.812
Dispersion	3	952.14	317.38	69.275
Condition time	3	37.13	12.378	2.702
Centrifugal speed	3	22.81	7.603	1.659
Centrifugal time	3	145.01	48.34	10.551
Error	0			
Total	15	1374.42		100

Table 11: ANOVA results of L16 for (CEC) value using (TSPP).

Source of variation	Degree of freedom	Sum of squares	variance	P contribution
Solid concentration	3	21.828	7.2761	11.662
Dispersion	3	151.391	50.464	80.884
Condition time	3	7.647	2.548	4.086
Centrifugal speed	3	4.288	1.429	2.291
Centrifugal time	3	2.015	0.672	1.076
Error	0			
Total	15	187.170		100

Table 12: Chemical composition of the beneficiated Ca-bentonite claystone sample.

SiO_2 %	Fe_2O_3 %	Al_2O_3 %	CaO %	MgO %	K_2O %	Na_2O %	SO_3 %	L.O.I %	Total %
48.32	5.97	18.53	2.15	3.05	0.35	3.20	1.10	10.42	93.30
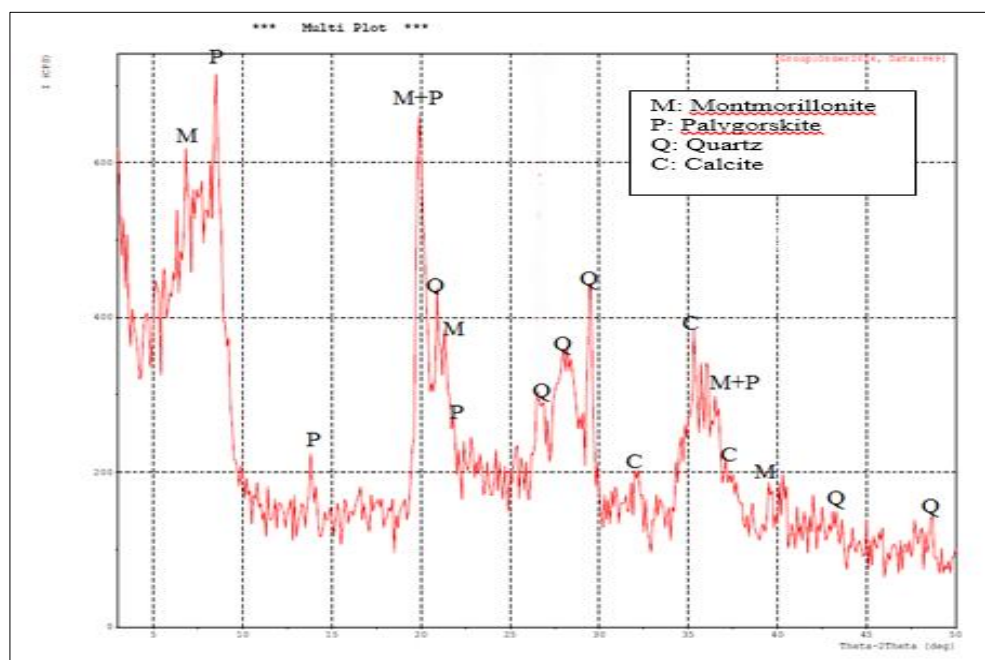
Figure 7: XRD - pattern of beneficiated clay.

4. Conclusions

The test results from the present work have shown that the dispersion sedimentation technique with tetrasodium pyrophosphate, as the dispersant, is very effective in beneficiating attapulgite-montmorillonite claystone from the crude claystone at a low slurry solid concentration, when addition (7 wt.% solid, 0.8 wt.% TSPP, 15 min conditioning time, 800 rpm centrifugal speed, 10 min centrifugal time). The dispersant quantity should be optimized in order to achieve good beneficiation results. It was found that tetrasodium pyrophosphate is much better than sodium tripolyphosphate as a dispersant.

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