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FULL LENGTH ARTICLE

Egyptian diatomite as high fluid loss squeeze slurry in sealing fractures and high permeable formation

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Abstract Lost circulation is the most costly mud related drilling problem, and induced fracture. Water slurry of diatomite is used as the high fluid loss squeeze slurry in the treatment of lost circulation and in decreasing fluid loss. Egypt has diatomite deposits, especially in El-Fayuom Depression. Fourteen samples were collected from Qasr El-Sagha at the northern shore of Birket Qarun. Samples were examined to identify the diatom species then subjected to X-ray fluorescence, XRD and grain size distribution tests. A total of 38 species related to 13 diatom genera were identified. *Cocconeis*, *Epithemia* and *Rhopalodia* were the predominant genera. The diatomaceous earth which acts as a filter aid material was tested with different additives; bentonite, lime, finely divided paper, polymer, barite and different concentrations with different types of lost circulation materials (LCM) to form a high fluid loss squeeze slurry. As a result the required time for collecting the filtrate was decreased to be in the range of 50 s to 1 min and 49 s comparing with the international standard which recommended the filtrate should be collected maximum within 2–3 min.

1. Introduction

Drilling fluid, or mud, may be defined as any fluid that is used in a drilling operation. A drilling fluid is circulated or pumped from the surface, down the drill string, through the bit, and back to the surface via the annulus. It is the single most important part of any earth excavation exercise, especially when drilling for oil and gas. Selection of the best fluid to meet anticipated conditions will minimize well costs and reduce the risk of catastrophes such as stuck drill pipe, loss of circulation, gas kick, etc [1–3].

Lost circulation is the term used to describe the condition characterized by lack of mud returning to the surface after being pumped down a well-bore. It is caused by the loss of some or the whole of the drilling fluid or mud at any depth into the natural fissures or due to high permeability of the formation or by pressure induced fractures [4,5], resulting in fluid loss and there are high costs associated with replacing drilling fluids [6]. Egyptian oil field production is distinguished by loss

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of circulation due to the rocky nature and the high porosity of Egyptian wells [7].

Many lost circulation materials are added to drilling fluids. One of the lost circulation water base mud (WBM) additives that is considered environmentally friendly is silica. Silicates are non-toxic and are multi-functional materials that are being used in drilling mud formulations by causing changes in density, ionic strength, and charge. Silica is needed for critical drilling mud functions such as: drill-bit cooling, bit cleaning, effective cuttings removal to surface, downhole pressure control and Shale stabilization [8].

Diatomite can be combined with other materials such as organic fibbers to be used as a lost circulation control material in water drilling fluids. It is typically used as a rheological modifier or processing aid in a "squeeze" treatment, it has a highly permeable nature which allows it to rapidly form a plug to bridge fractures. Diatoms produce a highly porous mineral which can be used to seal wide fractures, as it will lose fluid very rapidly to provide a solid plug on which an impermeable filter cake may then form. Silica constitutes about 80–90% of the chemical composition of the diatom frustule. Typical high fluid loss slurry contains a mixture of diatomite, bridging agents and barite suspended in either water or oil [9,10].

Almost any type of mud can be used as a carrying fluid for spotting lost circulation materials, but fluids that have extremely high filtration rates are best. Once the initial bridge forms, filtration allows the filtrate to be lost from the slurry depositing a firm filter cake within the fracture itself. This technique should be used against all types of loss zones. There are slight differences in application to these different loss zones, but the main distinction is increasing the size of the bridging agent as the loss zone becomes more severe [11].

The first high solids high fluid loss squeeze was performed with diatomaceous earth in 1956 by Carl Huber, (Phillips Petroleum Company) and John Crocker (Magcobar) on a Phillips Petroleum Company well north of Pampa, Texas. Drilling Specialties Company then a division of Phillips introduced the first commercial product Diaseal M® LCM, in 1964. Today several companies have altered the formula slightly and present it as something new to the industry [12].

This study focused on an environmentally friendly additive diatomite and the efficiency of this slurry. The collected Egyptian diatomite was examined in order to select the highest silicate samples to use in drilling as a high fluid loss squeeze slurry in sealing fractures.

2. Materials and methods

Fourteen sites were chosen to collect fossil diatomite samples from Qasr El-Sagha located at the west of the El-Fayoum depression 90 km southwest of Cairo. Locations of the sites were geographically determined in latitude and longitude by using the global positioning system (GPS).

Samples were prepared by the method described by Jouse et al. [13] and the slides were mounted using the method described by Proschkina–Laverenko et al. [14]. The diatom samples were counted according to the counting method described by Vilbaste [15]. Diatomite samples were prepared for scan electron microscope (SEM) according to Hasle and Fryxell [16].

The material was analyzed using X-ray fluorescence (XRF) by The Ministry of Petroleum, The Egyptian Mineral Resources Authority (EMRA). Centre Laboratory Sector (XRF Lab). The material was analyzed using the XRD test. Particle size distribution of the sample was determined according to API RP 13C [17].

Normal field tests were carried out on water based mud. They cover the properties that are most important to the Mud Engineer at the location. These are primarily performed to keep a record of the basic physical requirements of the mud (filtration, density, viscosity, gel strength, pH), with additional tests to monitor the performance of high-filter-loss-slurry squeeze [17–21].

The hydrostatic pressure that a column of mud exerts upon any point in the hole can be calculated according to [18] as follows:

Hydrostatic pressure (psi) = 0.052 × mud weight (ppg "pounds per gallon") x depth (ft.)

Rheological properties were determined according to:

a – Bingham plastic model

It was estimated according to [18] by the following equations:

$$\text{Plastic Viscosity PV (cP)} = \theta_{600} - \theta_{300}$$

$$\text{Apparent Viscosity AV(cP)} = \frac{\theta_{600}}{2}$$

$$\text{Yield Point Y.P.(lb/100 ft}^2) = \theta_{300} - \text{PV}$$

b – Relation between shear rate, shear stress and effective viscosity

They were estimated by the following equations:

$$\text{Shear Rate } \gamma\ (\text{s}^{-1}) = R \times 1.7034$$

$$\text{Shear Stress } \tau\ (\text{lb/100 ft}^2) = 1.0678 \times \theta$$

$$\text{Effective Viscosity E.V. (cp)} = \frac{300 \times \theta}{R}$$

c – Power low model

It was estimated by the following equations:

$$\tau = k\gamma^n$$

$$\text{Log}\tau = \log k + n \log \gamma$$

where, θ, viscometer dial reading at Speed R, R, rotary speed, k, consistency, n, flow index.

3. Results and discussion

3.1. Diatom diversity

This study shows that, from the collected 14 sites a total of 13 genera and 38 species were identified belonging to Bacillariophyta. The distribution of different diatomite species varied from one site to another but the six most common species are predominant in most sites (Table 1). It was observed that *Cocconeis placentula* var. *euglypta* is predominant in almost all sites and it frequently presents in only 5 sites. It occupies 17% of the total number of individuals counted at all sites followed by *C. placentula* which represented 16% of the total number of individuals. *C. placentula* is predominant in about 8 sites and frequently presents in 6 sites. *Epithemia sorex*, *Epithemia turgida*, *Rhopalodia vermicularis* and *Rhopalodia gibba* var *ventricosa* are predominant in about 5 sites and frequently presents in 9 sites except *R. gibba* var *ventricosa* which frequently presents in 6 sites and was common in 3 sites. *E. sorex*, *E. turgida* and *R. vermicularis* represented 13% of the total number of individuals while *R. gibba* var *ventricosa* represented 11% of the total number of individuals (Table 1) (Fig. 1).

Fig. 2 showed that the total number of identified genera and species were thirteen and thirty-eight respectively. The

Table 1 The mean frequencies of diatomite samples collected from different sites from Qasr El-Sagha located at the west of El-Fayoum depression.

Taxa	Sampling sites													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Division: Bacillariophyta														
Genus*: Amphora* Ehrenberg														
Amphora coffeiformis (Ag.) Kütz	–	–	+	+	–	–	–	–	+	*r*	–	–	–	–
A. ovalis Kütz	+	–	+	–	–	–	+	+	–	–	–	+	–	–
Genus: *Anomoneis* Pfitzer														
Anomoneis sphaerophora (Kütz) Pfitzer	+	+	–	–	–	–	–	–	–	–	–	–	–	–
Genus*: Aulacoseira* Thwaites														
Aulacoseira granulata (Ehr.) Simonsen	*r*	*r*	*r*	*c*	*c*	*c*	*c*	*r*	*r*	*r*	*r*	*c*	*c*	*c*
A. granulata var. *angustissima* (O.F. Müller) Simonsen	*r*	*r*	*r*	*c*	*r*	*r*	*r*	*r*	*r*	*r*	*r*	*r*	*r*	*r*
Genus*: Cocconeis* Ehrenberg														
Cocconeis placentula Ehr	*p*	*p*	*p*	*f*	*p*	*f*	*f*	*f*	*f*	*p*	*p*	*p*	*p*	*f*
C. placentula var.euglypta (Ehr.) Cl	*p*	*p*	*p*	*f*	*p*	*f*	*f*	*f*	*f*	*p*	*p*	*p*	*p*	*p*
Genus*: Cyclotella* Kützing														
Cyclotella kützingiana Thwaites	*c*	*c*	*c*	*r*	–	–	–	*r*	*r*	*r*	–	–	–	–
C. stelligera	*r*	*r*	*r*	*r*	–	+	–	–	+	–	–	–	–	–
C. meneghiniana Kütz	–	+	–	–	–	–	–	–	–	–	–	–	–	–
C. ocellata Pant	*r*	*r*	*r*	–	+	+	–	–	–	+	+	+	–	–
Genus*: Cymatopleura* W.Smith														
Cymatopleura elliptica (Bréb.) W.Sm	*r*	*r*	*r*	–	–	–	–	–	–	–	–	+	+	–
C. solea (Bréb.) W.Sm.	+	+	+	–	–	+	+	+	+	–	–	–	–	–
Genus*: Cymbella* Agardh														
Cymbella affinis Kütz	*r*	*r*	*r*	*c*	*r*	*r*	*r*	*r*	–	–	–	*r*	*r*	*r*
C. laencelata (Ehr.) V. H.	*r*	*r*	*r*	*c*	–	–	*r*	*r*	*r*	*r*	–	–	–	–
C. minuta Hilse	*r*	*r*	*r*	–	–	–	–	–	–	–	–	–	–	–
C. turgida (Greg.) Cl	–	–	–	+	–	–	–	+	–	–	–	–	–	–
C. tumida (Bréb.) V. H	*c*	*c*	*c*	–	–	*r*	*r*	*r*	*r*	–	–	+	+	–
C. ventricosa (Kütz.)	*r*	*r*	*r*	*c*	–	*c*	*c*	*c*	*c*	*r*	*r*	*r*	*r*	*r*
Genus*: Epithemia de* Brébisson														
Epithemia adanta (Kütz.) Bréb	*r*	*r*	*r*	–	–	–	–	–	–	–	–	–	–	–
E. sorex Kütz	*p*	*p*	*p*	*p*	*p*	*f*	*f*	*f*	*f*	*f*	*f*	*f*	*f*	*f*
E. turgida (Ehr.) Kütz	*p*	*p*	*p*	*p*	*p*	*f*	*f*	*f*	*f*	*f*	*f*	*f*	*f*	*f*
Genus*: Gomphonema* Hustedt														
Gomphonema constrictum var capitatum	*r*	*r*	*r*	–	–	–	–	–	–	–	–	–	–	–
G. parvulum (Ehr.) Cl	+	+	+	–	–	–	–	–	–	–	–	+	+	–
G. gracile (Ehr.)	+	+	+	–	–	–	–	+	+	–	–	–	–	–
G. intricatum Kütz	+	+	+	–	–	–	–	–	–	–	–	–	–	–
G. longiceps Ehr	–	–	–	+	+	–	–	–	–	–	–	–	–	–
Genus*: Navicula* Bory														
Navicula gastrum (Ehr.) Kütz	–	+	–	–	–	–	+	–	–	+	–	–	–	–
N. pupula var. capitata Skv. & Meyer	–	–	–	–	–	–	+	–	–	–	–	–	–	–
Navicula radiosa Kütz	+	+	*f*	*f*	*c*	–	–	*c*	–	*c*	–	–	–	–
Genus*: Rhopalodia* O. Müller														
Rhopalodia gibba (Ehr.) O.Müller	*c*	*c*	*c*	*f*	*c*	*c*	*c*	*c*	*c*	*c*	*c*	*c*	*c*	C
R. gibba var ventricosa (Ehr.) O.Müller	*f*	*f*	*p*	*p*	*p*	*p*	*p*	*f*	*f*	*f*	*c*	*c*	*c*	F
R. musculus (Kütz.) O.Müll	*c*	*c*	*c*	*c*	*r*	–	–	–	*r*	*r*	*r*	*r*	*r*	–
R. vermicularis O.Müller	*f*	*f*	*p*	*p*	*f*	*f*	*f*	*p*	*p*	*f*	*f*	*f*	*p*	F

(*continued on next page*)

Table 1 (*continued*)

Taxa	Sampling sites													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Genus: *Stephanodiscus* Ehrenberg														
Stephanodiscus astraea (Ehr.) Grun	*f*	*f*	*f*	*c*	*c*	*c*	*c*	*c*	*f*	*f*	*c*	*c*	*c*	F
S. astraea var. minutulus (Kütz.) Grun	*c*	*c*	*c*	*c*	*c*	*r*	*r*	*c*	*c*	*c*	*r*	*r*	*r*	C
Genus: *Surirella* Turpin														
Surirella carboni	–	–	+	+	–	–	+	–	–	–	–	+	–	–
S. ovalis Bréb	+	+	+	–	–	–	–	–	+	+	–	–	–	–
Total number of genera	13	13	12	11	9	8	11	11	10	10	7	11	8	6
Total number of taxa	31	32	32	17	16	17	19	21	21	20	14	20	17	13

p = predominant (50–20%), *f* = frequent (20–5%), *c* = common (5–1%), *r* = rare (1–0.2%), + = noted and – = not noted.

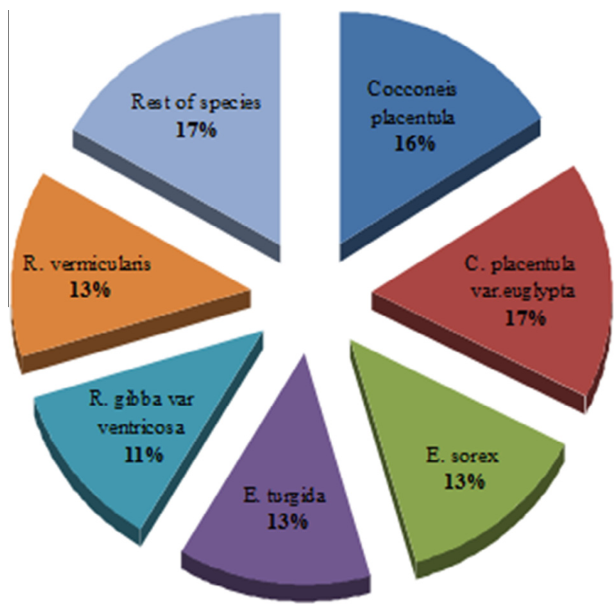

Figure 1 The Percentage of the most common 6 species (total No. of individuals counted/200 valve).

highest number of species was recorded in sites two and three, thirty-two, followed by site one which was represented by thirty-one species. Site fourteen was represented by the least number of genera and species. It was represented by only six genera and thirteen species. Sites eight and nine recorded 21 species followed by sites ten and twelve which recorded 20 species. Site seven was represented by 19 species. Sites four, six and thirteen were represented by 17 species. Number of species recorded in sites five and eleven was 16 and 14 respectively.

Cocconeis was the dominant genus followed by *Rhopalodia* then *Epithemia*. These genera were frequently found also with *Stephanodiscus* then *Cymbella* and *Navicula*. The least noted genus was *Anomoneis*, it was just noted twice in sites one and two. *Surirella* was noted in 8 sites. (Figs. 3 and 4)

C. placentula var. *euglypta*, *C. placentula*, *E. sorex*, *E. turgida*, *R. vermicularis* and *R. gibba* var *ventricosa* are normally heavily silicified and robust; their valves have thickened internal costae (Figs. 5 and 6) [22–26] .These species represent the most predominant individuals in the diatomite deposits, collected from Qasr El-Sagha located at the west of El-Fayuom Depression. Although *Aulacoseira granulata* was found in low amounts than the predominant species it was also highly silicified (Fig. 6). This analysis indicates that the diatomite deposits have high quality and are promising when used as a high fluid loss squeeze slurry in the treatment of lost circulation and in decreasing fluid loss.

3.2. X-ray fluorescence test (XRF)

By analyzing the material with X-ray fluorescence (XRF) the results in Fig. 7, showed that SiO_2 was the highest percent in the sample (41.64%) due to the presence of diatomite followed by CaO, Al_2O_3, Fe_2O_3 with percents equal to 18.39, 5.97, 4.04 respectively due to the presence of mud. The sample contains traces of Na_2O, K_2O, TiO_2, P_2O_5 with 1.93%, 0.65%, 0.36%, 0.18% respectively. These results were relatively in accordance with those of Nayak and Singh [27] who collected samples from India where SiO_2 had the highest percent [29] followed by Al_2O_3, Fe2O3 and CaO with 14.1%, 1.2% and 0.18% respectively. Na_2O and K_2O had 2.55% and 0.38% respectively but it doesn't contain any TiO_2 or P_2O_5. In the

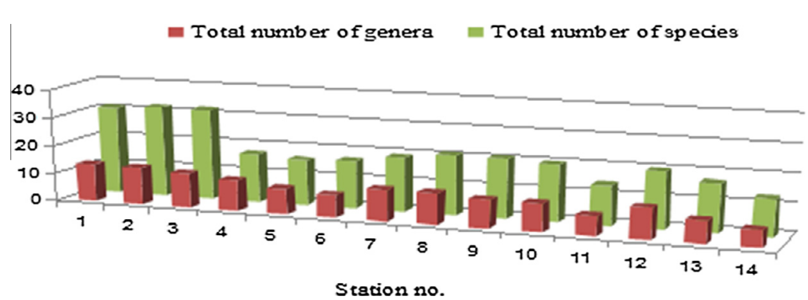

Figure 2 Number of genera and species for the fourteen sites.

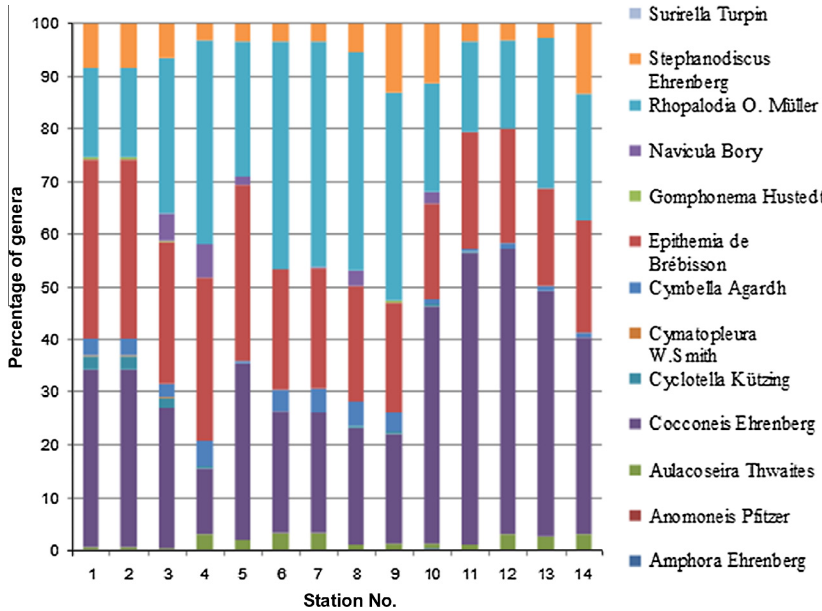

Figure 3 The Proportions of thirteen genera in fourteen sites.

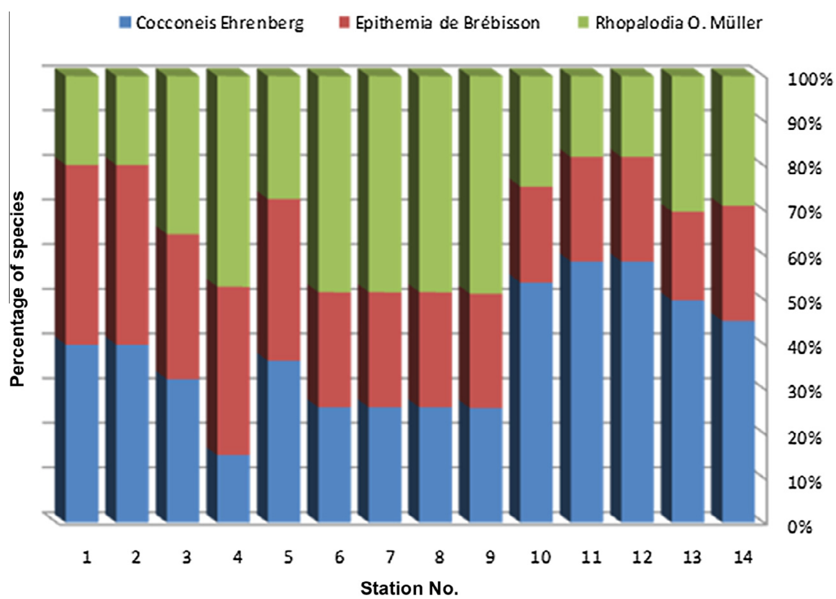

Figure 4 Proportions of the most common 3 genera in fourteen sites.

diatomite sample collected from Thailand it was reported that SiO_2 had 71.9% followed by CaO, Al_2O_3, Fe_2O_3 with percents equal to 0.17, 14.6 and 5.8 respectively. K_2O and TiO_2 had 1.95% and 0.51% respectively but it doesn't contain any Na_2O or P_2O_5 [28].

3.3. Elements of the X-ray diffraction test (XRD)

XRD pattern of crystalline materials shows sharp peaks while that of amorphous materials shows broad peaks (Fig. 8). A crystal is periodically arranged in a 3D space. On the other hand amorphous materials do not possess that periodicity and atoms are randomly distributed in 3D space. The scattering of X-rays by atoms is the point to be considered in that case. When there is a periodic arrangement of atoms the X-rays will be scattered only in certain directions when they hit the formed lattice planes (formed by atoms). This will cause high intensity peaks (the width of the peaks depends on other variables). If a certain amount of amorphous impurity is present, then the diffraction peak will be surrounded by slightly broad peaks from the amorphous phase. Amorphous phase: X-rays will be scattered in many directions leading to a large bump distributed in a wide range (2 Theta) instead of high intensity narrower peaks [29].

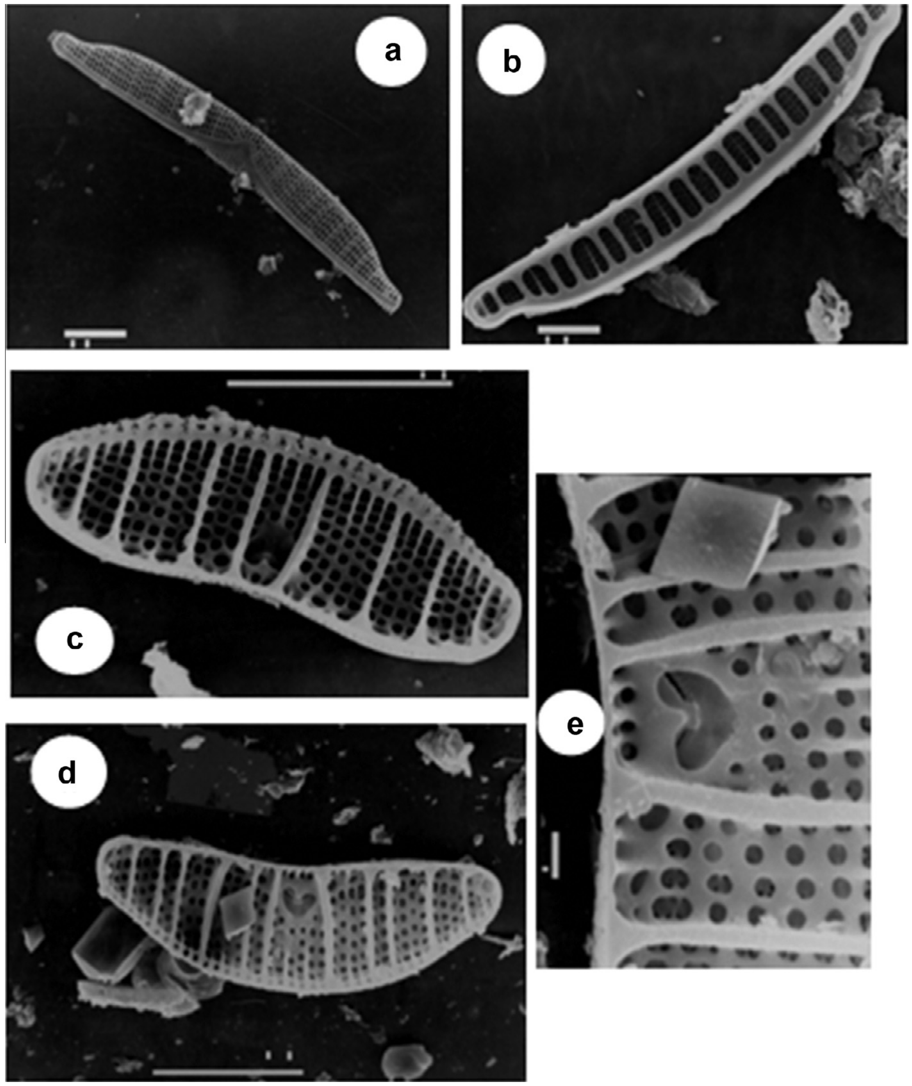

Figure 5 Scanning electron microscope images of some characteristic diatoms present in different sites from Qasr El-Sagha. (a and b) *Epithemia turgida*. (a) External view of the valve face (scale bar = 10 μm). (b) Internal view of the valve face showing robust internal costae (scale bar = 10 μm). (c–e) *Epithemia sorex*. (c and d) Internal view of the valve showing thickened internal costae (scale bar = 10 μm). (e) Magnified part of the valve view (scale bar = 2 μm).

3.4. The grain size distribution test

The particle size distribution in a given sample was described along with the help of grading curves. The grading curve for the obtained data is displayed in Fig. 9. The steep slope of the grading curve, which is from D to E (i.e. between 212 and 150 μm size), indicates the greater proportion of these size particles in the sample. The uniform slope of the grading curve, which in this case is from C to D (i.e. between 300 and 212 μm), E to F (i.e. between 150 and 106 μm) and F to G (i.e. between 106 and 75 μm), indicates continuous grading. Therefore, particles of sizes between 300 and 75 μm size are present maximum in the sample. Flat slope of the grading curve, which is from H to I (i.e. between 63 and 53 μm) and from I to J (i.e. between 53 and 45 μm), indicates lesser/negligible contribution of the particles of that size.

3.5. API tests for the high-filter-loss-squeeze slurry

3.5.1. Effect of diatomite and bentonite

The formula of the high-filter-loss-squeeze slurry was first tested to be formed from diatomite and bentonite (Fig. 10). The diatomite can function as a filter aid material with a high fine solid to assist in forming a permanent seal of the permeable formation. Nagre et al. [30] reported that, bentonite swells considerably when exposed to water, makes it ideal for protecting subterranean formations from invasion of drilling fluids. Bentonite is widely used as a mud additive for viscosity improvement.

The fastest complete loss of filtrate was (1:25 min.) while using 20 ppb diatomite without any addition of bentonite. The time increases with increasing the quantity of bentonite. The filter cake thickness was 13.3, 10.7, 7.4 and 3.6 mm using diatomite only with concentrations of 80, 60, 40 and 20 ppb.

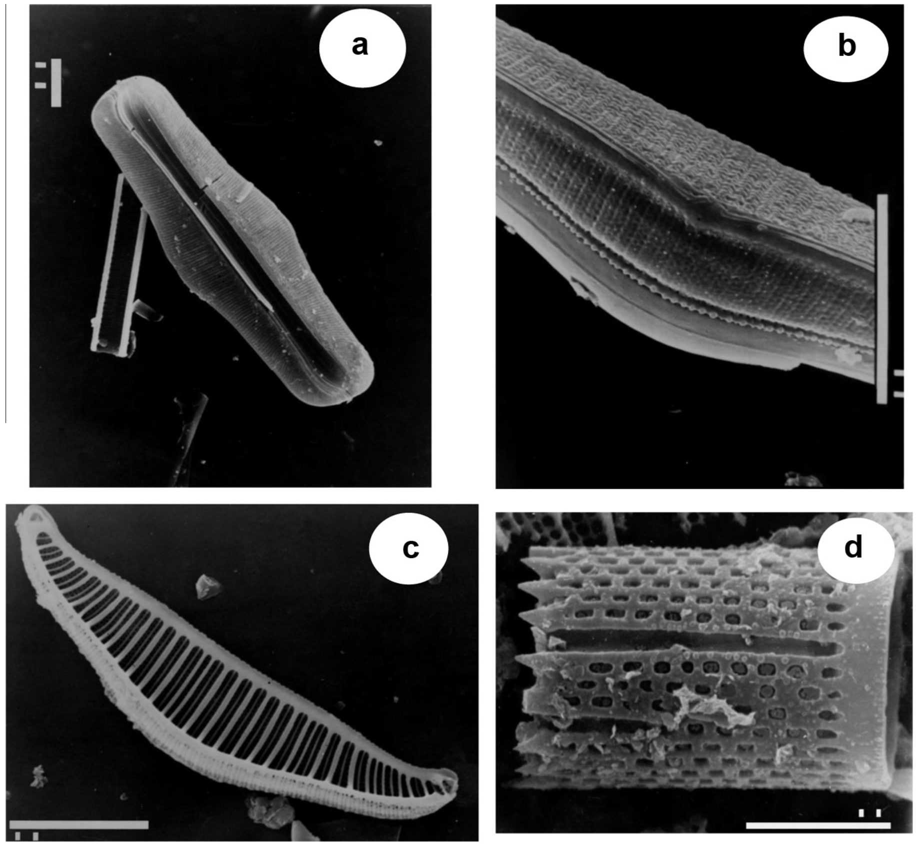

Figure 6 Scanning electron microscope images of some characteristic diatoms present in different sites from Qasr El-Sagha. (a–c) *Rhopalodia gibba* var *ventricosa*. (a) Girdle view of the frustule (scale bar = 2 μm). (b) External view of the valve face showing the heavy silicification (scale bar = 10 μm). (c) Internal view of the valve showing thickened internal costae (scale bar = 10 μm). (d) External view of *Aulacoseira granulata* showing the dense rows of mantle areolae (scale bar = 10 μm).

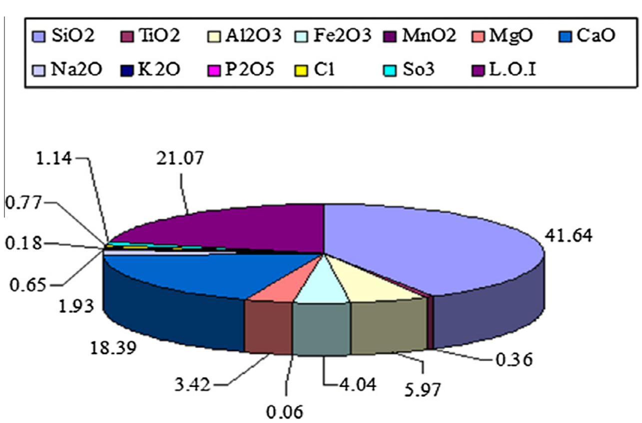

Figure 7 Chemical composition of natural diatomaceous earth sample by X-ray fluorescence (XRF).

Cake thickness was increased because the sample lost its all filtrate so all its solid components were precipitated causing an increase in the thickness of the filter cake. The filter aid properties of diatomite were stopped against this high concentration of bentonite. The best concentration was 40 ppb diatomite and this is due to that the cake in the other concentrations had many cracks which indicated that this cake will not firmly close the fracture in the reservoir during drilling. Kilchrist and Verret [31] illustrated that the amount of diatomite employed in the HAC (Hardenable alkaline composition) preferably should comprise at least 22.5 percent by weight of the said composition and a better concentration is in the range from about 22.5 to about 50 percent by weight. An especially preferred level is from about 24% to about 35 percent by weight. Results of this experiment showed that the addition of bentonite had a passive effect on high filter loss squeeze slurry especially during increasing its concentration on both the time for filter loss and mud cake thickness.

3.5.2. Effect of lime

Lime is believed to react with diatomite and with sodium silicate, if present, to form calcium silicate and sulfate. It increases the compressive strength of the cake. Lime was added to diatomite without the addition of any bentonite. The preferred amount of lime can range from about 15 to about 20 ppb, and most preferably was 15 ppb. The time for total filtrate losses was 1:50 min. for both 15 and 20 ppb of lime (Fig. 11). The thickness of the mud cake was 9.2 and 8 mm for lime concentrations of 15 and 20 ppb respectively so the selected concentration was 15 ppb lime. Lime can range from about 9 to about 50 weight percent. Better results with lime range from about 22.5 to about 35 weight percent and the best from about 24 to about 35 percent by weight of the mixture [31].

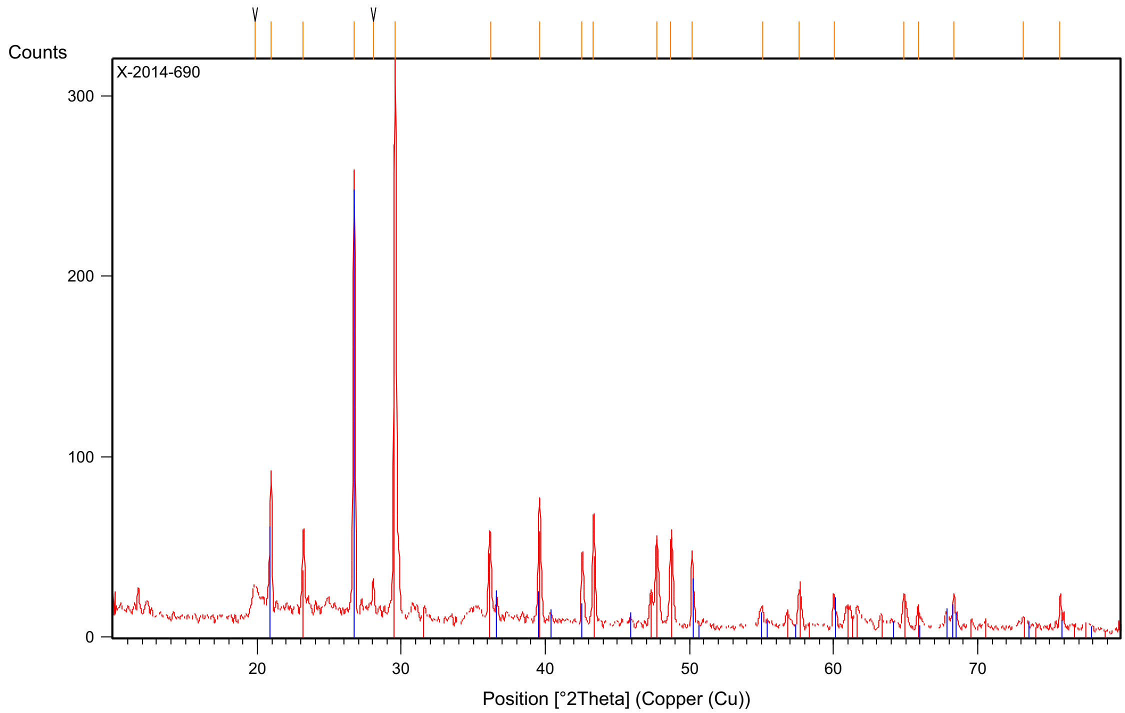

Figure 8 The XRD for untreated samples with start position [°2Th.] 10.0100, end position [°2Th.] 79.9900, step size [°2Th.] 0.0200, scan step time [s] 0.8000, scan type continuous and K-alpha1 [Å] 1.54060.

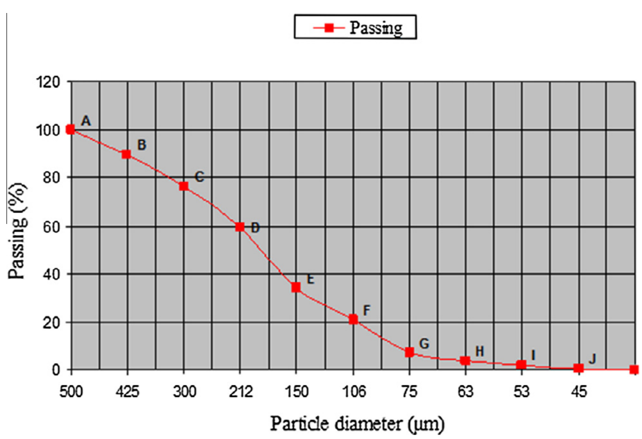

Figure 9 Grading Curve shows the particle size distribution of diatomaceous earth.

3.5.3. Effect of hay

Agriculture waste was used as it was considered as a local cheap material to form the function of Fibrous materials. It is the first time hay to be used as a fibrous material in drilling fluid generally and it is not used in high-filter loss squeeze slurry. Hay forms a mat over the fracture to reduce its size as a step in forming the filer cake. Many authors used agricultural waste materials, for example, rice straw, sugarcane bagasse, saw dust, cotton stables [32,33], orange mesocarp [34], weeds, *Eichoria crassipes* [35].

In the present study, different concentrations of hay were tested for enhancing the property of high-filter loss squeeze slurry. The best concentration was 3 ppb. The filter cake thickness was increased to reach 13.5 mm in 1:36 min (Figs. 11 and 12). Hay concentration more than 3 ppb causes it to float on the surface without interfering with cake structure. The time was decreased and cake thickness was increased but the homogeneity of the filter cake was still poor. It was very important to use another fibrous material to enhance the homogeneity of the filter cake.

3.5.4. Effect of paper

Finely divided paper was used as a suspending agent for the fluid in aqueous slurries. The preferred amount of paper can range from about 3 to about 7.5 ppb. The best level is to provide excellent suspending activity without unduly increasing the viscosity or unduly restricting water loss by 5 ppb (Figs. 11 and 12). The homogeneity of the filter cake was enhanced by mixing paper with the aqueous phase to form a paper pulp as suspending material.

Kilchrist and Verret [31] mentioned that the amount of paper employed to stabilize the slurry is at least 2 percent by weight of the composition of HAC (Hardenable alkaline composition). The amount of paper can range from about 2 percent by weight to about 30 percent by weight. A preferred range is from about 3.5 to about 15.5 percent by weight, the more preferred level is about 90% to about 13 percent by weight to provide excellent suspending.

3.5.5. Effect of polymer

Addition of polyacrylamide (synthetic cross-linked polymer) was tested in range from 0.25 to about 3 ppb. The filter cake thickness increased to about 19.6 mm and the filtrate was completely lost in only 0:50 s (Figs. 11 and 12). The homogeneity of the filter cake was enhanced. It will readily swell following

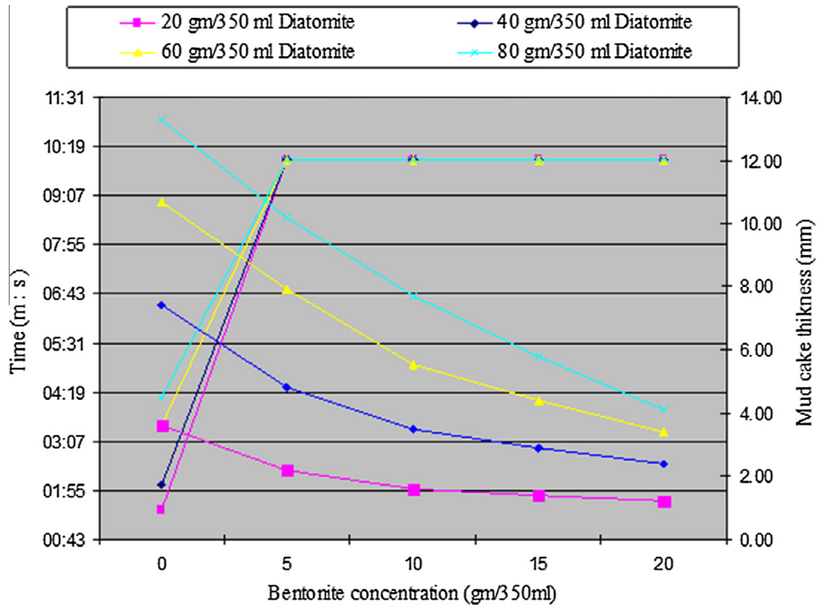

Figure 10 Effect of different concentrations of bentonite and of diatomite on time of API filter press and filter cake thickness.

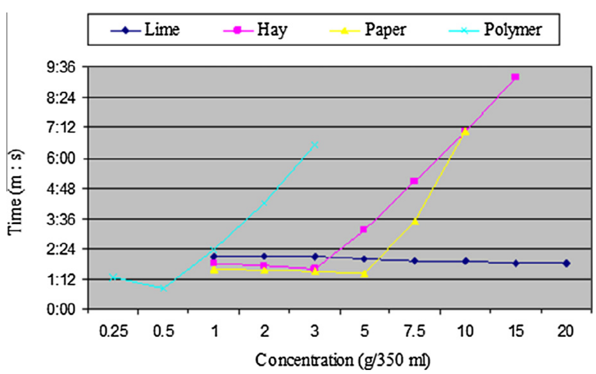

Figure 11 Effect of different concentrations of hay, lime, paper and polymer on filtration time using API filter press at an optimum concentration of diatomite.

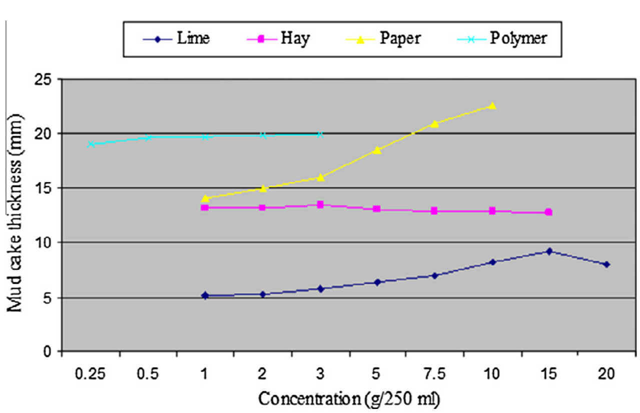

Figure 12 Effect of different concentrations of hay, lime, paper and polymer on mud cake thickness at optimum diatomite concentration.

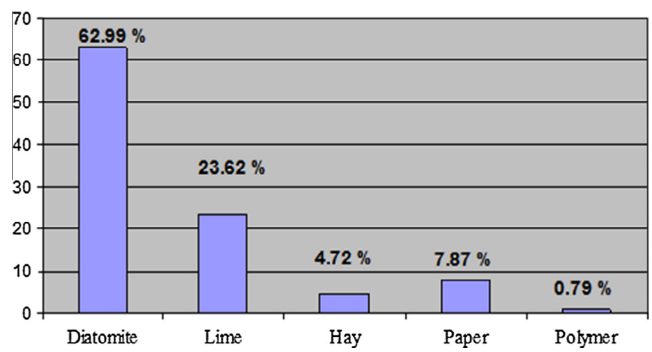

Figure 13 Percent of mixed material forming a high filter loss squeeze slurry.

exposure to water or aqueous based fluids forming a kind of soft gel. Polyacrylamide is a non-toxic polymer. It is used for water-based drilling fluids, which can effectively increase and stabilize viscosity.

Fig. 13 showed the Percent mixed material forming a high filter loss squeeze slurry. Burts [36] illustrated water base mud comprising water, soluble cross linkable agent which is chromic carboxylate, polymer and diatomite. Composition includes 4% polymer, 1% cross linking agent and 95% diatomite, or 24% polymer, 6% cross linking agent and 70% diatomite, or 35% polymer, 10% cross linking agent and 55% diatomite. The amount of filter aid to be utilized is generally not dependent upon the amount of polymer or cross linking agent, but rather, is that amount sufficient to form a plug to retain the polymer in place until it cross links sufficiently to remain in a place of its own.

3.5.6. Effect of barite on the slurry

Weighting Materials are substances with a high specific gravity which can be added to the mud to increase its density, usually

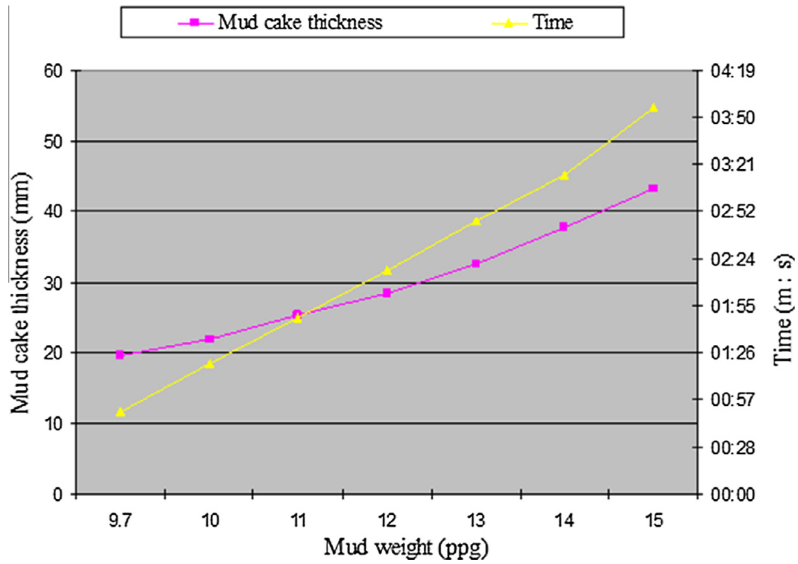

Figure 14 Effect of different mud weights on mud cake thickness and time in fresh water mud.

Table 2 Effect of 40 g/350 ml diatomite, 15 g/350 ml lime, 3 g/350 ml hay and 5 g/350 ml paper with different concentrations of LCM on API filter press and filter cake.

$CaCO_3$, g/350 ml	Mica, g/350 ml	Total filtrate volume, cm^3	Total time, min	Mud cake thickness, mm
0	0	275	00:50	19.6
	5	268.2	01:00	20.38
	10	256.8	01:10	21.2
	15	246	01:21	22.1
5	0	266	01:03	20.4
	5	261.2	01:13	21.2
	10	250.8	01:21	22.07
	15	240.9	01:31	23.09
10	0	259.8	01:16	21.1
	5	253.3	01:27	22.07
	10	244.7	01:34	23.04
	15	236.4	01:41	24.01
15	0	250.8	01:28	22.5
	5	244.7	01: 38	23.4
	10	236.4	01:42	24.32
	15	227.3	01:49	25.3

to control formation pressure. Barite (Barium Sulfate with the chemical formula of $BaSO_4$) is by far the most common weighting material used in drilling fluids. The weighted slurry should have about the same density as the drilling mud in use. To prevent blowout, the uncontrolled flow of fluid from the formation into the well, the drilling fluid must have a density effective to provide a greater pressure than that exerted by the formation fluids. However, densities must not be too high, because excessive hydrostatic pressure can cause further loss of circulation [37]. Results in Fig. 14 show that by increasing mud weight the total filtrate time increases until it reached its maximum of 3:56 min at a mud weight of 15.

3.5.7. Effect of lost circulation materials on the slurry

The selection of an appropriate bridging material is more critical during work over operations. Calcium carbonate (acid-soluble) and mica with different sizes and concentrations were used (Table 2). Addition of LCM with different sizes and concentrations was in accordance to [38] which determines the type of loss as follows:

1. For seeping lossAdd different LCM sizes and shapes to obtain a 10- to 20-lb/bbl total concentration of LCM.
2. For partial lossAdd a combination of different LCM sizes and shapes to obtain a 15-lb/bbl total concentration of LCM.

Table 3 Effect of the slurry composition on rheological properties.

Mud weight	9.7	10	11	12	13	14	15
θ_{600}	38	40	46	55	65	79	94
θ_{300}	24	28	33	39	46	59	72
θ_{200}	18	20	26	30	32	41	54
θ_{100}	11	13	17	20	22	26	33
θ_{6}	3	4	6	8	9	12	15
θ_{3}	2	2	3	4	5	7	10
Bingham plastic parameters							
Gel 10 s (lb/100 ft^2)	3	5	5	6	7	9	11
Gel 10 min (lb/100 ft^2)	5	7	8	9	11	12	16
Thixtropy (lb/100 ft^2)	2	2	3	3	4	3	5
Plastic viscosity (cP)	14	12	13	16	19	20	22
Apparent viscosity (cP)	19	20	23	27.5	32.5	39.5	47
Yield point (lb/100 ft^2)	10	16	20	23	27	39	50
LSYP (lb/100 ft^2)	1	0	0	0	1	2	5
Power low parameters							
Flow index (n)	0.663	0.621	0.569	0.572	0.596	0.576	0.555
Consistency index (lb.s^n/100 ft^2)	0.384	0.539	0.89	1.045	1.045	1.461	2.015

3. For complete lossAdd a combination of different LCM sizes and shapes to obtain 20- to 30-lb/bbl total concentration of LCM.

3.5.8. Effect of the slurry on rheological properties

Rheology refers to the deformation and flow behavior of all forms of matter. It is defined as its internal resistance "friction" generated by a fluid when a force is applied to cause it to flow. This internal friction, a result of the attraction between molecules of liquid, is known as shear stress; the greater the resistance, the greater the viscosity. When force is applied to cause a fluid to flow, the resultant effect is known as shear rate. It is defined as velocity gradient across adjacent fluid layers while in laminar flow. Rheological fluids measurements help in determining how this fluid will flow under a variety of different conditions. This information is important in the design of circulating systems required to accomplish certain desired objectives in drilling operations [39].

The investigated structure of the high fluid loss squeeze slurry enhances the rheological properties of the drilling fluid. From the obtained results shown in Table 3 the rheological property increases by increasing the mud weight. The lowest rheological property was obtained without the addition of bentonite while a mud weight of 15 was the highest rheological property. It is obvious that, polymer and paper could be used as a good viscosifier because they raise the gel strength of the bentonite water suspension.

Fig. 15 showed that plastic viscosity, apparent viscosity, and yield point were 14 cP, 19 cP and 10 lb/100 ft^2 without the addition of barite and reached its maximum of 22 cP, 47 cP and 50 lb/100 ft^2 respectively at a mud weight of 15. According to Clear [40] plastic viscosity and yield point were 14 cP and 13 lb/100 ft^2 respectively in a sample containing diatomite, paper, lime with no barite. Plastic viscosity and yield point reached 13 cP, 52 lb/100ft^2 at a mud weight of 14 and 15 cP, 66 lb/100 ft^2 at a mud weight of 16.

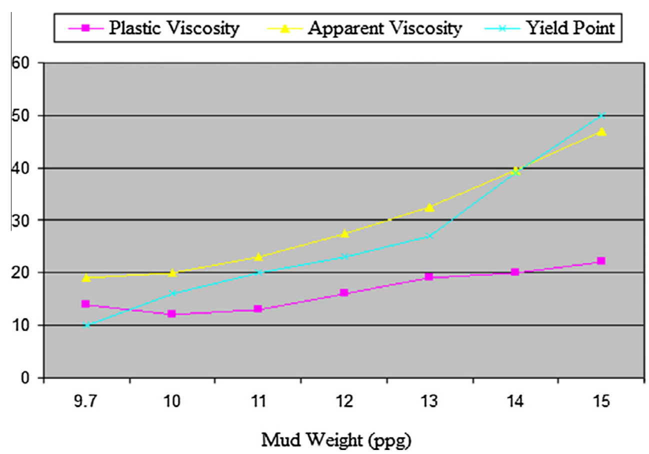

Figure 15 The rheological properties of water base drilling fluid.

The pattern of the curve obtained from the shear rate and shear stress relationship showed that the shear rate and shear stress were related in a nonlinear manner as shown in Fig 16. This nonlinear relationship showed that the mud obeyed the power law model for non-Newtonian fluid. In addition, the nonlinear relationship between the shear rate and shear stress showed that the mud is pseudo-plastic. According to Alderman et al. [41], a fluid is pseudo-plastic when the consistency curve obtained from the shear rate and shear stress relationship passes through the origin and is nonlinear. It was observed from Table 3 that the flow index, n, is less than 1 for each type of mud with the various studied mud weights. According to Mewis et al. [42], a fluid for which the n value is less than 1 is said to have a pseudo-plastic flow behavior.

Fig. 17 showed that the relation between the effective viscosity (cP) versus shear rate (s^{-1}) for the investigated mud weights was decreasing the effective viscosity while increasing the applied shear rate.

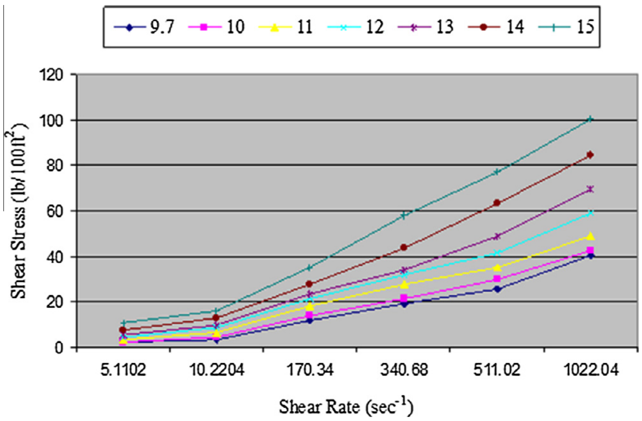

Figure 16 The relation between shear rate and shear stress for water base mud with different mud weights (ppg).

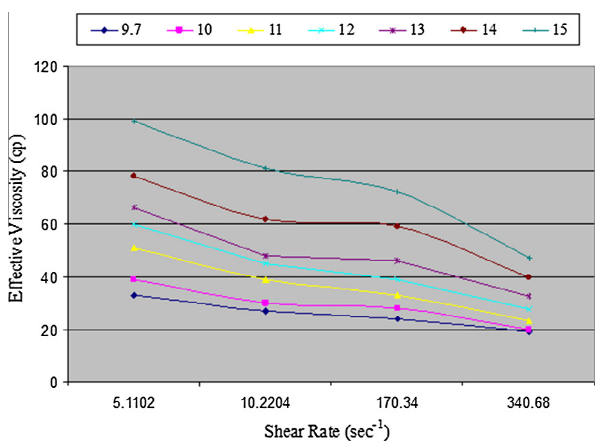

Figure 17 Relation between shear rate and effective viscosity for water base mud with different mud weights (ppg).

4. Conclusion

Egyptian diatomite can be used as a high fluid loss squeeze slurry for the treatment of lost circulation treatment and in decreasing fluid loss as it contains diatoms species which were heavily silicified. By adding different additives as bentonite, lime, finely divided paper, polymer and barite to the diatomaceous earth the required time for collecting the filtrate was decreased to be in the range of 50 s to 1 min and 49 s comparing with the international standard which recommended the filtrate should be collected maximum within 2–3 min.

References

[1] ASME, in: American Society of Mechanical Engineers, Shale Shaker Committee, Gulf Publishing Company, Burlington, USA, 2005, pp. 15–26.
[2] R. Gardner, Ann. Occup. Hyg. 47 (3) (2003) 201–210.
[3] HWU, Drilling Engineering, Lecture Material, Institute of Petroleum Engineering, Heriot Watt University, Edinburg, UK, 2009 (Unpublished) pp. 358–363.
[4] D. Vollmar, V. Wittig, R. Bracke, Geothermal Drilling Best Practices: The Geothermal Translation of Conventional Drilling Recommendations – Main Potential Challenges IGA Academy Report 0104, 2013.
[5] A.A. Oluwatoni, A.A. Sarah, U.A. Julius, Int. J. Eng. Technol. 4 (2) (2014) 109–117.
[6] J. Finger, D. Blankenship, Handbook of Best Practices for Geothermal Drilling, Sandia Nationa Laboratories Albuquerque, New Mexico 87185 and Livermore, California, 2010.
[7] M.I. Abdou, Formation Damage Caused by Oil-Well Drilling Fluids, Lulu Publishing, 2014.
[8] A.M. Al-Sabagh, M.R. Noor El-Din, H.M. Mohamed, J. Disper Sci. Technol. 30 (7) (2009) 1079–1090.
[9] B.D. Burts, Conformance Improvement Additive, Conformance Treatment Fluid Made Therefrom, Method of Improving Conformance in a Subterranean Formation, Patent No. US 2006, 6, 997, 261 B2.
[10] A.M. Al-Sabagh, M.I. Abdou, H.E. Ahmed, A.A. Aboulrous, Egypt. J. Petrol. 22 (4) (2013) 481–491.
[11] A.M. Al-Sabagh, M.I. Abdou, A.A. Khalil, H.E. Ahmed, A.A. Aboulrous, Egypt. J. Petrol. 23 (1) (2014) 27–34.
[12] Drilling Specialties Company Lost circulation Guide, 2015, pp. 1–81. <www.drilling spec ia lties.com>.
[13] A.P. Jouse, A.I. Proschkina-Laverenko, V.C. Sheshykova, Diatom Analysis, vol. I, Pub. "Geol. Liter", Leningard, 1949, 339pp.
[14] A.I. Proschkina-Laverenko, S.I. Gleser, A.P. Jouse, I.V. Makarova, V.S. Schesschykova-Poretzaja, The Diatoms of USSR. Fossil and Recent, vol. I, Pub. House "Sov. Nauke", Leningard Branch, 1974, 372 pp.
[15] S. Vilbaste, in: D. Marino, M. Montresor (Eds.), Proceedings of the 13th International Diatom Symposium, Biopress Limited, Bristol, 1994, pp. 259–269.
[16] G.R. Hasle, G.A. Fryxell, Trans. Am. Microsc. Soc. 89 (1970) 469–474.
[17] API RP 13C Recommended Practice 13C on Drilling Fluid Processing Systems Evaluation, December 2004, fifth ed., ISO 13501, API RP 13C Compliance Definitions, Clarity, and FAQs, 2004.
[18] API 13B-1, Recommended Practice 13B-1 for Field Testing Water based Drilling Fluids ISO 10414-1: 2008 (Identical), Petroleum and Natural Gas Industries – Field Testing of Drilling Fluids – Part 1: Water based Fluids.
[19] API 13B-2, Recommended Practice 13B-2 for Field Testing of Oil-based Drilling, Fluids, fourth ed., March 2005 Petroleum and Natural Gas Industries – Field Testing of Drilling Fluids – Part 2: Oil-based Fluids.
[20] API 13I, Recommended Practice 13I for Standard Procedure for Laboratory Testing Drilling Fluids, seventh ed., February 2004 ISO 10416: 2002 (Modified), Petroleum and Natural Gas Industries – Drilling Fluids – Laboratory Testing.
[21] API 13J, Recommended Practice 13J for Testing of Heavy Brines, fourth ed., May 2006 ISO 13503-3:2005, (Identical) Petroleum and Natural Gas Industries – Completion Fluids and Materials, Part 3: Testing of Heavy Brines.
[22] J.C. Kingston, in: John D. Wehr, Robert G. Sheath (Eds.), Freshwater Algae of North America, vol. 16, Elsevier Science, USA, 2003, pp. 559–636.
[23] F.E. Round, R.M. Crawford, D.G. Mann, The Diatoms. Biology and Morphology of the Genera, Cambridge Univ. Press, Cambridge, UK, 1990, 747p.
[24] J.C. Marks, R.L. Lowe, Can. J. Fisheries Aquat. Sci. 50 (1993) 1270–1278.
[25] R.L. Lowe, in: R.J. Stevenson, M.L. Bothwell, R.L. Lowe (Eds.), Benthic Algal Ecology in Freshwater Ecosystems, Academic Press, San Diego, 1996, pp. 57–76.
[26] R.L. Lowe, in: John D. Wehr, Robert G. Sheath (Eds.), Freshwater Algae of North America, vol. 19, Elsevier Science, USA, 2003, pp. 669–684.
[27] Preeti Sagar Nayak, B.K. Singh, Bull. Mater. Sci. 3 (30) (2007) 235–238 (Indian Academy of Sciences).

[28] A. Chaisena, K. Rangsriwatananon, J. Sci. Technol. 4 (11) (2004) 289–299.
[29] A.Q. Selim, A.A. El-Midany, S.S. Ibrahim, in: A. Méndez-Vilas, J. Díaz, (Eds.), Microscopy: Science, Technology, Applications and Education, 2010, pp. 2174–2181.
[30] R.D. Nagre, P.A. Lin Zhao, Owusu, J. Petrol. Coal. 56 (3) (2014) 222–230.
[31] M.J. Kilchrist, R.J. Verret, Method of Decreasing Lost Circulation During Well Operations and Additive Therefor, Patent No. 7, 297,663 B1, 2007.
[32] A. Hebeish, N.Y. Abou-Zied, A. Waly, A. Higazy, Cellulose Chem. Technol. 22 (1984) 591–605.
[33] V.K. Varshney, S. Naithani, Chemical Functionalization of Cellulose Derived from Nonconventional Sources" Green Chemistry and Technology, India, 2011, pp. 43–60.
[34] O. Akaranta, L.C. Osugi, Cellulose Chem. Technol. 31 (1997) 195–198.
[35] B.K. Barai, R.S. Singhal, P.R. Kulkarni, Carbohydr. Polym. 32 (1997) 229–231.
[36] B.D. Burts, Lost Circulation Additive, Lost Circulation Treatment Fluid Made the Refrom, and Method of Minimizing Lost Circulation in a Subterranean Formation, Patent No. US 20040023815 A1, 2004.
[37] M.F. Strachan, Studies on the Impact of a Water – Based Drilling Mud Weighting Agent (Barite) on Some Benthic Invertebrates", Submitted for the degree of Doctor of Philosophy, Heriot-Watt University, 2010.
[38] Fann Instrument Company, Lost Circulation, Bridging Materials and the PPA, 2007, pp. 1–2. <www.fann.com>.
[39] M.I. Abdou, H.E. Ahmed, Petrol, Sci. Technol. 28 (9) (2010) 934–945.
[40] E.E. Clear, Method Using Lost Circulation Material for Sealing Permeable Formations, Patent No. US 4369844, 1983.
[41] N.J. Alderman, D.R. Babu, T.L. Hughes, G.C. Maitland, 4th International Congress on Rheoological, Sydney, vol. I, 1988. p. I.
[42] J. Mewis, J.F. Willaim, A.S. Trevor, W.B. Russel, J. Chem. Eng. Res. Develop. ALCHE 19 (1989) 415.