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Rheological behavior of sepiolite suspensions homogenized by ultra-turrax high-speed homogenizer

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Abstract: The ability to disperse sepiolite suspensions is of great interest in various fields such as the production of cosmetics, pharmaceuticals, colors, etc., and in preparing the sample material for analytical purposes. In this study, the flow curves and time-dependent rheological behavior of sepiolite aqueous suspensions homogenized by Ultra-Turrax high-speed rotor-stator homogenizer were investigated under operating conditions. Forward and backward flow curves for homogenized sepiolite suspensions at different homogenization times and solid concentrations were plotted, and the amount of existing hysteresis loop was measured to determine the best conditions for the obtention of a suitable product having gel-forming properties for industrial and/or analytical applications. The results showed that the dispersed sepiolite suspensions exhibited a substantial increase in the viscosity values owing to gel formation, and thixotropic behavior that was rapidly improved by the increasing the solid content of suspension/ homogenization time, and their apparent viscosity decreased by the increasing the time of shearing, and flow characteristic was time-dependent. However, changing the distance between dispersing tool/bottom showed no favorable effect on preserving the rheological behavior of sepiolite suspensions, although the apparent viscosity decreased marginally due to increased distance between dispersing tool/bottom. Also, two time-dependent/independent models, namely the Power-law model and the Weltman model, were used to describe the thixotropy behavior, and different parameters of these models were analyzed. The Weltman model was found to be appropriate to fit the time-dependency behavior of sepiolite suspensions and the most obtained value for model parameters *A* and *B* belonged to sepiolite suspension with 6 wt% at 25 min, which was considered as the optimized sample in the applied experiments, its final viscosity level increased from 0.82 to 12.55 Pas.

Keywords: sepiolite, rheology, modeling, ultra-turrax homogenizer

1. Introduction

Among the commonly used fibrous clay minerals, sepiolite has known quick application growth essentially because of its outstanding sorption capacity and rheological properties. A wide range of technological applications such as drilling fluids, emulsion paints and coatings, bitumen anionic emulsions, liquid animal feeds, mortars, liquid suspension fertilizers, adhesives, and processing aids are based on the remarkable rheological properties of sepiolite (Álvarez et al., 2011). The sepiolite fibres, which formed bundles-like aggregates by surface interaction between individual needle-type particles, are separated into individual needles which show a high-thixotropic behavior in water with high apparent viscosity at low shear when they are dispersed in water or other liquid systems. External mechanical shearing is necessary to disaggregate the crystal bundles of sepiolite and enhance the dispersion of sepiolite rods, leading to suspensions of high viscosity and with rheological properties that are highly dependent on the purity of sepiolite, particle size and shape, suspension preparation, concentration, pH, ionic strength, and so forth. It is defined as the dispersion degree determined by the energy applied to the system, provided that it keeps the fibers combined in bundles to overcome the forces such as electrostatic, hydrogen bonding, and Van der Waals among the rods. Particularly, a high-speed shearing causes this energy because of strong shearing, impact, and dispersion forces on the suspension along the tangent to the rotor. Moreover, the bulk bundles are torn up into smaller bundles

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or single rods, making them effectively micronized, mixed, and dispersed quickly. This method has been mostly used to obtain uniform and stable nanoparticle suspensions having better rheology for the production of healthcare, medicine, and electronic parts; to produce micronized waxes dispersions for the ink, toner, and coatings, and the pigment or dye-based solid inks; to manage the wet milling process for active pharmaceutical ingredients (APIs); to disorder cells in fermentation recovery processes, and even to maintain the polymorph transformation during the crystallization of pharmaceutical products (Zhang and Li, 2012) In the case of processing of clay minerals, high-speed stirring is usually used for the preparation of colloidal clay suspensions (Wang and Wang, 2016).

The degree of sepiolite dispersion and hence the properties of the resultant rheology of sepiolite suspensions due to change or modification in surface property depend mainly on the mixing conditions such as mixer shear rate and time, types of mixer used, presence or not of later shearing processes, conditioning temperature, suspension pH, ionic strength, etc. used in the application process. In sepiolite-based rheological applications, different processing techniques are employed to disperse sepiolite in water or other liquids including simple mechanical mixing (Tunç et al., 2008; Tunç et al., 2011; Tanrıver et al., 2015), ultrasonication mixing (Simonton et al., 1988; Zheng et al., 2011; Chemeda et al., 2014; Abdo et al., 2016; Liu et al., 2020), high shear mixing (Santaren et al., 1987; Viseras et al., 1999; Çınar et al., 2009; Altun et al., 2010). To the best of our knowledge, almost no research has focused on evaluating the feasibility of high-speed shearing on the rheological behavior of sepiolite suspensions prepared/sheared by a high-shear rotor/stator homogenizer at different parameters such as speed, homogenization time, the distance between dispersing tool/bottom, etc.

The present study aimed to examine the effect of high-shear homogenization with Ultra-Turrax high-speed homogenizer based on the rotor-stator principle at different parameters (homogenization time, suspension solid concentration, the distance between dispersing tool/bottom) on the rheological behavior of suspensions consisting of fibrous sepiolite with Turkish origin. Finally, some empirical equations, which can model the suspension rheology of sepiolite, were investigated.

2. Materials and methods

2.1. Materials

Brown-colored sepiolite sample was received from AEM Co., Sivrihisar region of Turkey, and crushed below 2 mm as a starting material by adapting a series of crushers, including jaw and roll crushers. The sample was not treated or purified by any chemical methods before and after the crushing. A sieve analysis (dry) by using a Retsch AS200 sieve shaker was performed to determine the particle size distribution of the crushed sample (Fig. 1). The results indicated that the particle size distribution of the sample ranged from 0.425 to 2 mm; and the mean (d_{50}) was about 0.85 mm.

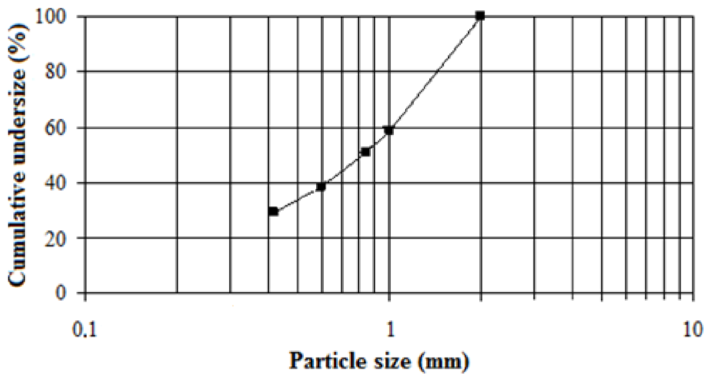

Fig. 1. Particle size distribution of the sample crushed below 2 mm

2.2. Methods

The mineralogical and chemical compositions of the sepiolite sample were determined by X-ray diffractometer (XRD) (Panalytical X Pert Pro MPD, PANalytical B.V., Almelo, The Netherlands) and X-ray fluorescence (XRF) spectrometer (ZSX Primus II XRF, Rigaku Corporation, Tokyo, Japan). The BET surface area, pore volume, and average pore diameter of the sepiolite sample were measured with a Quantachrome surface area analyzer (Quantachrome, Ltd., Hook, Wales, UK) using N_2 gas adsorption at –196ºC. Before the measurement, the sample was outgassed for 10 h by heating at 60ºC under a vacuum.

The zeta potentials of sepiolite samples were determined at 23 ± 5°C by Zetasizer Nano Z (Malvern Instruments), which uses micro-electrophoresis/electrophoretic light scattering technology. The zeta potential measurements were carried out as a function of the pH for natural sepiolite suspensions. In this procedure, the sepiolite suspensions with a minimum solid concentration of 0.1 g/dm^3 to avoid particle interaction (10^{-2}% mass according to Malvern's recommendation) were conditioned at 500 rpm. The pH value of each suspension was adjusted by adding either NaOH or HCl. The pH of the solution was measured using the combination HI 1131 pH electrode with Ag/AgCl reference cell (HI 2210 pH meter, Hanna Instruments). After the conditioning for 10 min, followed by centrifuging for another 10 min at 3000 rpm an aliquot was taken from the supernatant and used for the zeta potential measurements. The average of ten measurements was taken to represent the measured potential. The mean relative standard deviation (σ) of the values reported usually did not exceed 5.

The sepiolite suspensions at three different solid concentrations (4 wt%, 5 wt%, and 6 wt%) were homogenized at 15000 rpm using the Ultra-Turrax high-speed homogenizer based on the rotor-stator principle (IKA Ultra-Turrax T25, Works Inc., Wilmington, NC, USA) with dispersing tool S25 N-18G (Stator diameter: 18 mm; Rotor diameter: 12.7 mm) setting its distance from the beaker bottom to 10, 15, 20, 25, and 30 mm at various homogenization times (5, 10, 15, 20, 25, and 30 min). Using the rotor-stator principle, the Ultra-Turrax is a high-performance dispersing machine to produce suspensions in batch operations. The ability to apply intense shear and shorten homogenization cycles and the emergence of new variations on the original rotor/stator mixer concept gives this homogenizer broad appeal for applications that require immiscible fluids to be formulated into emulsions or agglomerated powders to be dispersed into a liquid medium.

The sepiolite suspensions were allowed to stand, cooling to room temperature (25°C) for 5 min after each homogenization cycle. Subsequently, their rheological measurements were conducted using a rotational rheometer (RheolabQC, Anton Paar GmbH, Graz, Austria) with a temperature control bath which works according to the Searle principle considered in this investigation because it is the one that was used in generating the rheological properties of the slurries in the slurry database used. Specifically, the CC39/ QC-LTD concentric cylinder measuring system (according to DIN EN ISO 3219 and DIN 53019) was selected for this work. The apparent viscosity of 6 wt% sepiolite suspensions was measured, taking one (1) reading at regular intervals for 20 sec while the spindle of the rheometer was rotating at 5 rpm, keeping the temperature constant at 25°C. Particular care was taken to keep the pH values of all sepiolite suspensions lying between 8.5 and 9 since some studies indicate that interactions between fibers and silanol groups control the rheological properties (Simonton and Komarneni, 1988), and sepiolite suspension exhibited its highest viscosity at pH of 8–9 (Çınar et al., 2009).

Searle principle (Fig. 2): A cup is matched with a so-called spindle that is placed in the sample. The speed on the viscometer is preset, and the spindle starts to rotate. The sample in the cup follows this movement, and in further consequence, the torque (force) required for turning the spindle against the fluid’s viscous forces is measured. A spring connects the motor of the viscometer and the spindle. The rotation (torque) of the spindle deflects the spring. Optical sensors detect the deflection and, as a result, we get the sample's viscosity.

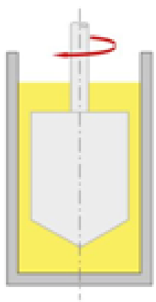

Fig. 2. Searle principle used in the study

The measured time-dependent flow properties of sepiolite suspensions were also modeled by the Power-law model and the Weltman model expressed as presented Eq. (1, 2) and Eq. (3), respectively:

$$\tau = K\,\gamma^n \tag{1}$$

where τ is the shear stress (pa), γ is the shear rate (s^{-1}), K is the consistency coefficient (pa s^n), and n is the flow behavior index (dimensionless).

The hysteresis loop as shown in Eq. (2) was obtained from the area between the upstream data and downstream data. This area was obtained by the difference between integrating the area for forward and backward measurements from γ_1 (initial shear rate) to γ_2 (final shear rate):

$$\text{Hysteresis loop area} = \int_{\gamma_1}^{\gamma_2} k\gamma^n - \int_{\gamma_1}^{\gamma_2} k'\gamma^{n'} \tag{2}$$

where k, k', and n, n' are the consistency coefficient and flow index behavior for forward and backward measurements, respectively.

$$\tau = A + B\ln(t) \tag{3}$$

where, τ is the shear stress (pa), t is the shearing time (s), A and B are constant parameters representing the initial stress and the time coefficient of structure breakdown, respectively, that characterize a material's time-dependent behavior.

3. Results and discussion

3.1. Mineralogical and chemical characterization

Fig. 3 illustrates the semi-quantitative mineralogical analyses by X-ray diffraction belonging to raw sepiolite, and its respective chemical analysis is provided in Table 1. The X-ray diffraction (XRD) pattern of the raw sepiolite sample (Fig. 3) revealed that the major component was sepiolite followed by dolomite. The intensities of dolomite peaks in the XRD along with a relatively high content of CaO (6.01%) of raw sepiolite (Table 1), suggest that the raw sepiolite used in this study was quite heterogeneous with a moderate proportion *(~53%)* of the main phase. While the ratio of SiO_2/MgO of the sample is about 1.91, its theoretical ratio is 2.23, and Tolsa S.A. sepiolite (Spain) has a range of 2.48–2.52 (Ünal and Erdoğan, 1998). Additionally, the BET specific surface area of the raw sepiolite sample was found to be 291 m^2/g. The high BET value of the sample can be attributed to its fibrous morphology, fine particle size, and most importantly the structural channels (or tunnels) which characterize sepiolite minerals. Based on these considerations, it can be concluded that the raw sepiolite having a high specific surface area has short fibers with closed textures and is consistent with the higher content of Si-OH groups.

Table 1. The chemical analysis of raw sepiolite

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5	SrO	SO_3	F	LOI*
%	46.40	0.93	0.38	6.01	24.10	0.25	0.54	0.06	0.01	0.08	0.08	1.51	19.67

*LOI: Loss on ignition

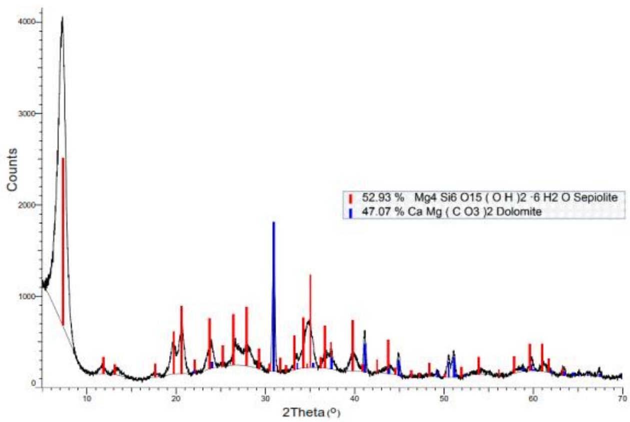

Fig. 3. X-ray diffraction profile of raw sepiolite

3.2. Effect of homogenization on rheological properties

Fig. 4 shows the shear rate against shear stress of 6 wt% suspensions of sepiolite under different homogenization times performed for 30 min over a 5-min interval. The shear rate first increased between 0 and 280 sec^{-1} and then decreased to its starting point at the same rate, and a hysteresis loop was obtained, which indicated that the sepiolite suspensions provided pseudoplastic, highly thixotropic (the area of the hysteresis loop of the flow curves), and suspending behavior at all homogenization times. This implies that the breakdown effect prevails the sepiolite suspension. The increasing of homogenization time resulted in a decrease in the shear stress values to reach a minimum at 5 min of homogenization time, and then these values increased gradually up to show a maximum at 25 and 30 min of homogenization time while the degree of their thixotropic behaviors is similar because their area of the hysteresis loop remained almost the same at all homogenization times (Table 2). Moreover, the change in the flow behavior of 6 wt% sepiolite suspension is minor at the homogenization time of over 25 min. This indicates that the 25 min shearing time is sufficient to fully disperse the sepiolite fiber bundles (Fig. 4), which could form large clusters and entrap more water into these clusters resulted in an increase in the viscosity of the system.

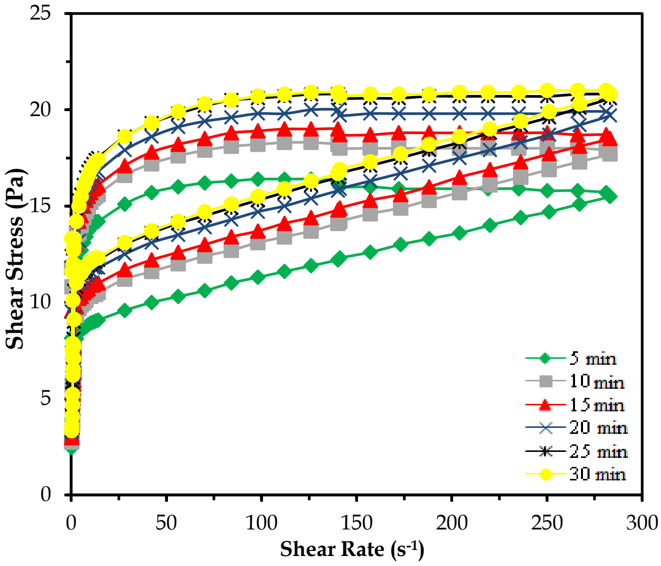

Fig. 4. Hysteresis loop of the flow curves of 6 wt% dispersed sepiolite suspension at different homogenization times (natural pH of 8.5, speed 15000 rpm, distance between dispersing tool/bottom 15 mm)

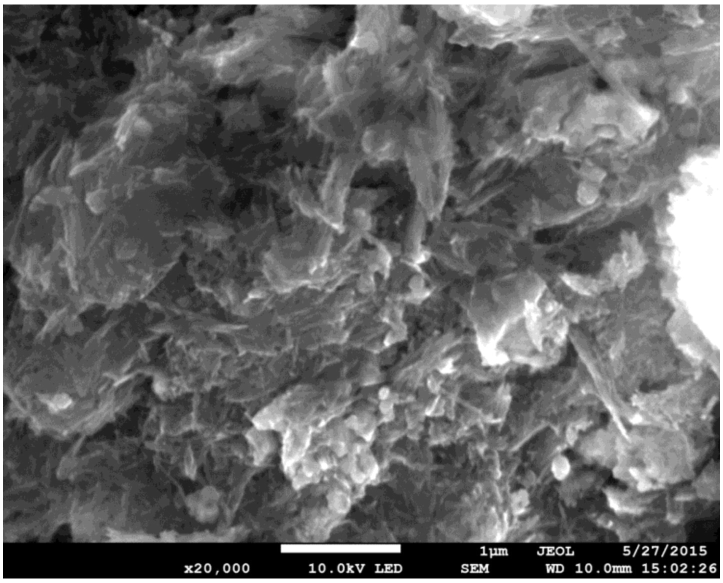

Fig. 5. SEM micrograph of raw sepiolite

Table 2. Hysteresis loop area for dispersed sepiolite suspensions at different experimental conditions studied

Solid Concentration (%)	Homogenization time (min)	Integrating area for up curve	Integrating area for down curve	Hysteresis loop area (Pas^{-1})
4	5	2027.91	1650.14	377.77
	10	2177.10	1811.14	365.96
	15	2297.50	1869.71	427.79
	20	2132.80	1826.46	306.34
	25	2331.37	1913.53	417.84
	30	2358.33	1932.90	425.43
5	5	2967.03	2362.73	604.27
	10	3341.35	2735.24	606.11
	15	3473.72	2765.53	708.19
	20	3919.02	3159.72	759.32
	25	3298.10	2578.05	720.05
	30	3665.40	3055.97	609.43
6	5	4604.59	3602.12	1002.47
	10	5029.26	4166.91	862.35
	15	5330.60	4362.49	968.11
	20	5606.89	4643.41	963.48
	25	5781.31	4849.79	931.52
	30	5731.87	4908.01	823.86

The zeta potential values of the sepiolite without homogenization (Fig. 6) were measured to study the influence of the surface charge density on capable of holding enough water molecules around them to increase the overall amount of structured water also supported this behavior. Figure 6 revealed that the isoelectric point (iep) of used sepiolite was 3.22, so its surface is a structure maker surface and can promote the structure of water (Healy and Fuerstenau, 1965).

The effect of homogenization time on the apparent viscosity of 6 wt% sepiolite suspensions (Fig. 7) showed that the apparent viscosity of sepiolite suspensions slightly decreased with a similar trend depending on increased shearing time and increases gradually as the homogenization time increases up to 25 min. In this view, maximum apparent viscosity (12.55 Pas) was observed at the homogenization time of 25 min. The viscosity values obtained with a homogenization time of 25 min were observed to increase by more than 1.5-fold than that of 5 min. Eventually, the increase stopped after 25 min of dispersion because homogenization over a certain period leads to the temperature increase of sepiolite

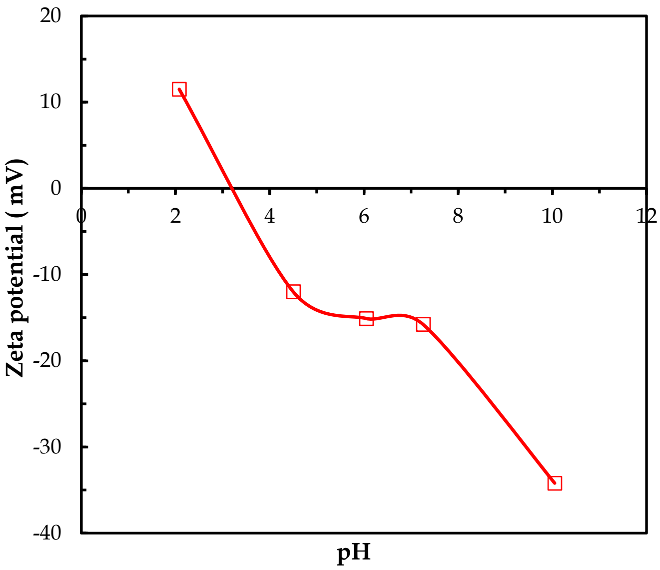

Fig. 6. The variation of zeta potential of raw sepiolite as a function of suspension pH

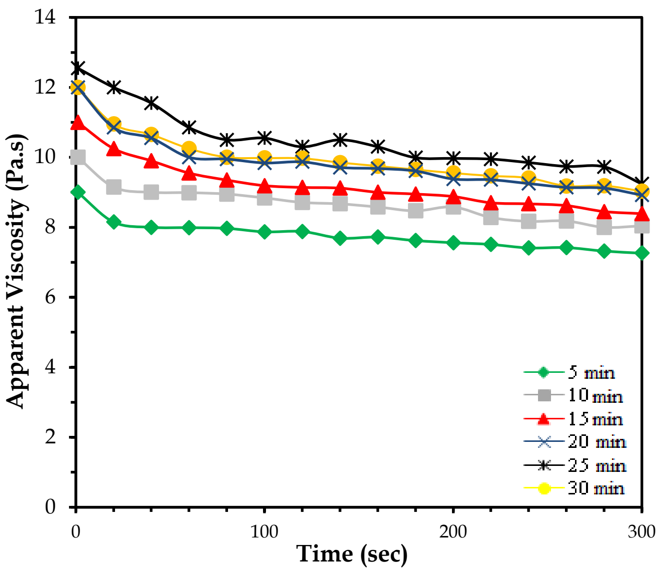

Fig. 7. Viscosity profiles of 6 wt% homogenized sepiolite suspension at different homogenization times as a function of shearing time (natural pH of 8.5, speed 15000 rpm, distance between dispersing tool/bottom 15 mm)

gel that significantly influences the viscosity of the suspension. When temperature increases, energy is supplied to the sample, hence intermolecular interactions and internal friction during flow reduce, therefore viscosity of the system decreases (Sharoba et al., 2005). Moreover, the increasing apparent viscosity of sepiolite suspensions can explain an increasing degree of fiber separation, indicated by the absence of fiber agglomerates and bundles, and the extent of random fiber orientation.

Figure 8 shows the rheogram of sepiolite suspensions for distances between dispersing tool/bottom ranging from 10 mm to 30 mm. All the plots (Fig. 9) indicate the thixotropic behavior of sepiolite suspensions. Shear stress increased by narrowing the distance. Nevertheless, the distance between dispersing tool/bottom has no favorable effect on preserving the rheological behavior of sepiolite suspensions, although the apparent viscosity decreases marginally due to increased distance between dispersing tool/bottom. The distance between dispersing tool/bottom of 15 mm that produces the max. apparent viscosity is considered optimum.

As is known, one of the factors that contribute to the non-Newtonian flow behavior of fluid is varying the solids concentration. Figure 10 presents the shear stress vs. shear rate profile for homogenized sepiolite suspension of varying concentrations. The presence of a hysteresis area between the curves representing shear stress versus shear rate data for increasing and decreasing shear rates indicates that the flow of the samples was time-dependent. From the figure above, it is clear that for the concentration of homogenized sepiolite suspension, the rheological behavior is drastically modified when >5%, and the flow profile of 6 wt% homogenized sepiolite suspension is indicative of high thixotropy. In contrast, the flow behavior character of homogenized sepiolite suspension with the concentrations of 4 and 5 wt% can be seen to be moderately thixotropic with some yield stress. A moderate thixotropic character can be seen in a slightly lower viscosity in the downward curve versus the upward curve of the graph. The areas of hysteresis loops for sepiolite suspensions (Table 2) confirm that the degree of thixotropy of 6.0 wt% sample is orders of magnitude greater than that of the 4.0 and 5 wt% samples. From this data, the shear stress increased as the concentration of sepiolite suspension increased because the particles were brought closer to each other, which restricts their movement; these particles tend to increase the shear stress or force necessary to maintain a given rate. As a result, the yield stress and plastic viscosity of the material increase due to the increased particle-particle interaction and random network structure strength, which cause the formation of a continuous gel structure, preventing their settling by gravity (very open structures composed of individual sepiolite fibers) instead of individual fibers that occur in lower concentrations.

The apparent viscosity profiles of homogenized suspension for three different concentrations of sepiolite are plotted as a function of the shear rate (Fig. 11). All the curves are quasi-parallel decreasing as the shear rate increases, the values corresponding to sepiolite concentration of 6 wt% being

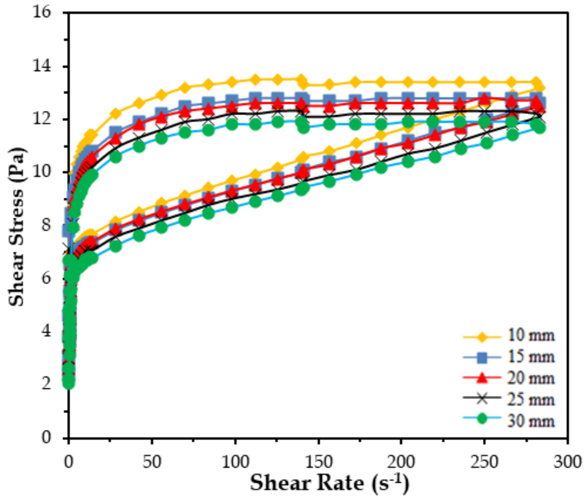

Fig. 8. Hysteresis loop flow curves of 5 wt% homogenized sepiolite suspension at different distances between dispersing tool/bottom (natural pH of 8.5, speed 15.000 rpm, homogenization time *20* min)

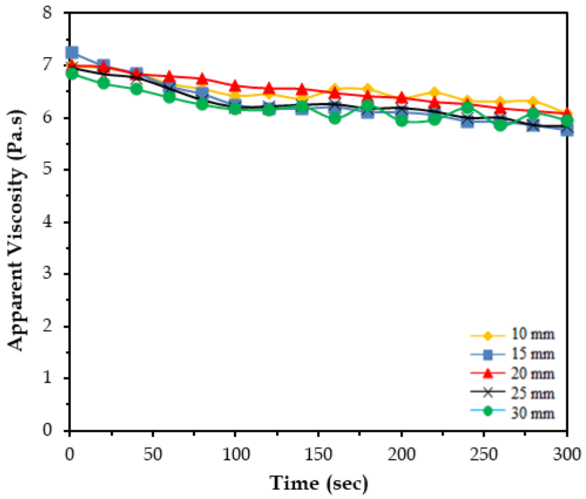

Fig. 9. Viscosity profiles of 5 wt% homogenized sepiolite suspension at different distances between dispersing tool/bottom as a function of shearing time (natural pH of 8.5, speed 15.000 rpm, homogenization time *20* min)

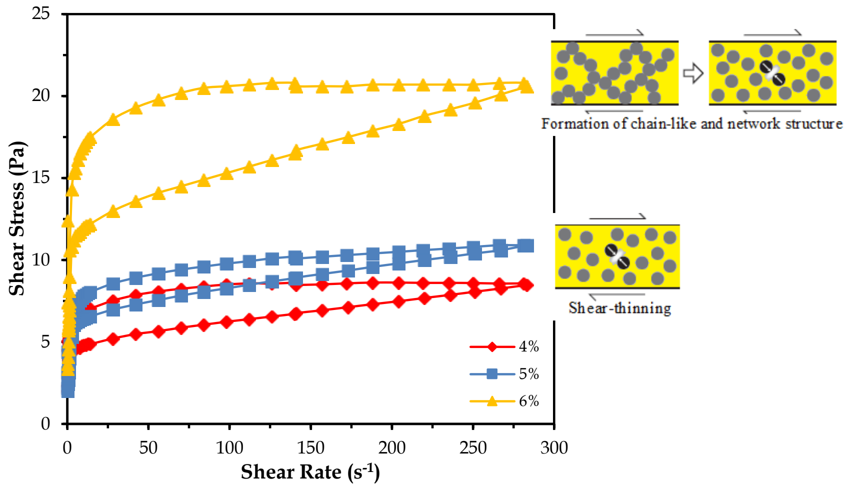

Fig. 10. Hysteresis loop flow curves of homogenized sepiolite suspensions for different solid concentrations (natural pH of 8.5, speed 15.000 rpm, homogenization time 25 min, distance between dispersing tool/bottom 15 mm)

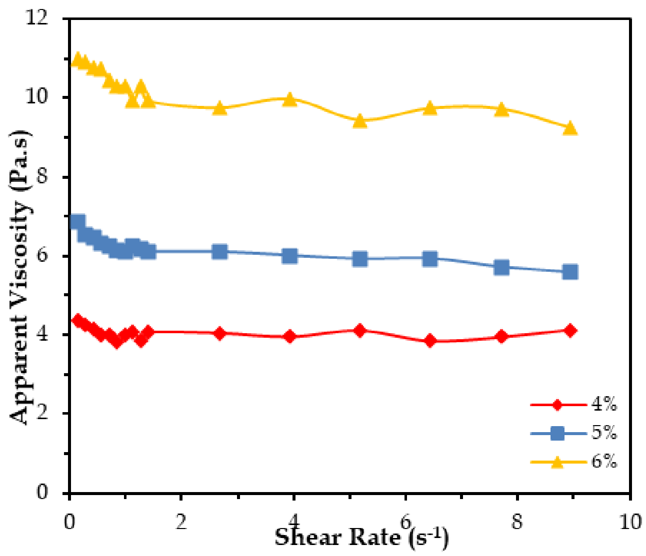

Fig. 11. Apparent viscosity vs shear rate for homogenized sepiolite suspension at different solid concentrations (natural pH of 8.5, speed 15.000 rpm, homogenization time 25 min, distance between dispersing tool/bottom 15 mm)

approximately 2.5 times those of the concentration of 4 wt%. In all cases, increasing the concentration of sepiolite suspensions leads to increased viscosity. This increment is less noticeable, and the fluid's molecular structure or network reaches a steady state at higher shear ratios where the intermolecular forces that are to build the structure/network and break it down by shear force are in equilibrium (no further decrease in viscosity). In fact, the shear forces are strong enough to disrupt the secondary bonds holding the sepiolite particles together at a very low shear rate, resulting in deformation or disruption of clusters/aggregates, which finally leads to a decrease in the apparent viscosity.

3.3. Time-dependent/independent rheological modeling

The knowledge of the rheological properties of suspensions is of paramount importance, and its correct measurement provides helpful information for the development of flow models for engineering applications, formulation of commercial production, design and process evaluation, quality control, storage stability, and the selection of the proper equipment (Bekkour et al., 2005). In this view, sepiolite suspensions have been an object of interest. The Power-law model (Eq. 1) and Weltman model (Eq. 3) were used to predict the variation of apparent viscosity with shear rate and the time-dependent non-Newtonian behavior of sepiolite suspensions homogenized by Ultra-Turrax high-speed homogenizer, respectively.

The Power-law model is used extensively to describe the non-Newtonian flow properties of liquids in theoretical analyses as well as in practical engineering applications (Barnes et al., 1989). Table 1 shows the fitting parameters of the Power-law model for the experimental data of the homogenized sepiolite suspensions at different homogenization times and solid concentrations, measured in the forward and backward directions. The relatively good R^2 values (≥ 0.90) obtained for all sepiolite suspensions considering the downward curves indicate that the Power-law model gave a good fit to the experimental data (Table 3), which was confirmed by Figs. 6 and 11. That is to say, the Power-law model appears to be only suitable for describing the flow behavior of homogenized sepiolite suspensions, as indicated by the high R^2 values measured in the backward direction. The K and n values increased slightly with the increase of the solid concentration of sepiolite suspensions and did not show any significant change with increasing the homogenization time at all solid concentrations. In that regard, the effect of the increasing homogenization time and solid content on K and n of the sepiolite suspensions is minor. Nevertheless, the n value less than one, an indicator for non-Newtonian flow and expressed a shear-thinning behavior, was the highest in concentrated sepiolite suspensions at given homogenization times. This means that the concentrated sepiolite suspensions are least susceptible to shear-thinning and most susceptible to supplied stress.

Table 3. The fitting parameters of the Power-law model

Solid concentration (%)	Homogenization time (min)	Forward direction			Backward direction		
		K (Pa s^n)	*n*	R^2	*K* (Pa s^n)	*n*	R^2
4	5	3.9078	0.1306	0.915	2.7651	0.1582	0.891
	10	4.5653	0.1128	0.967	3.2540	0.1457	0.905
	15	3.9811	0.1529	0.795	3.2505	0.1505	0.907
	20	4.9608	0.0909	0.979	3.3449	0.1396	0.931
	25	5.2239	0.0988	0.942	5.2239	0.0988	0.942
	30	4.8824	0.1155	0.934	3.5079	0.1415	0.917
5	5	4.4797	0.1817	0.725	3.2710	0.1980	0.908
	10	5.1647	0.1768	0.750	3.7020	0.2027	0.912
	15	3.8781	0.2444	0.803	3.8639	0.1961	0.917
	20	6.2371	0.1707	0.776	4.7490	0.1809	0.907
	25	4.1444	0.1887	0.879	3.7055	0.1902	0.926
	30	6.3969	0.1523	0.770	4.7207	0.1752	0.910
6	5	6.1657	0.2067	0.743	4.725	0.2092	0.912
	10	8.8403	0.1499	0.703	5.2514	0.2175	0.912
	15	7.7379	0.1899	0.764	5.6104	0.2133	0.914
	20	7.8624	0.1971	0.786	6.1736	0.2064	0.915
	25	9.5689	0.1625	0.771	6.3036	0.2110	0.923
	30	11.816	0.1164	0.792	6.5245	0.2064	0.910

Table 4 shows the analysis results according to the Weltman model for homogenized sepiolite suspensions of different concentrations and homogenization time. The Weltman model (Eq. 3) indicates the logarithmic decrease in shear stress with shearing time. In the Weltman model, parameter *A* indicates the flow resistance, having a close correlation with the yield stress, relating with the shear stress at the time 1s, and parameter *B* – the time coefficient of thixotropic breakdown – is "the product of the rate of breakdown of the thixotropic structure and time of agitation at a constant rate of shear" (Weltman, 1943). The *A* and *B* constants appeared in the range of 4.1577 to 13.1840 Pa and 0.125 to 0.575 Pa, respectively. It can be understood that increasing the solid rate and homogenization time has caused

Table 4. The fitting parameters of the Weltman model

Solid concentration (%)	Homogenization time (min)	*A* (Pa)	–*B* (Pa)	r^2
4	5	4.1577	0.188	0.76
	10	4.9590	0.264	0.94
	15	4.9509	0.192	0.90
	20	5.6479	0.319	0.76
	25	6.0373	0.379	0.81
	30	6.1982	0.339	0.90
5	5	6.2481	0.125	0.84
	10	7.1944	0.334	0.94
	15	7.7418	0.386	0.92
	20	8.3015	0.431	0.89
	25	9.0834	0.537	0.87
	30	9.5309	0.575	0.86
6	5	9.4034	0.340	0.83
	10	10.4661	0.380	0.81
	15	11.5180	0.495	0.86
	20	12.6032	0.586	0.87
	25	13.1840	0.608	0.85
	30	12.5583	0.557	0.87

a low increase in the magnitude of *A* and *B* (The increase in *A* value is more than that in *B* value). The increasing *A* value indicates an increased degree of fiber separation, which can explain the increasing viscosity of sepiolite gel with shear rate and shearing time at a constant shear rate. Also, the Weltman model was appropriate to fit the time-dependency behavior of sepiolite suspensions. The most obtained value for parameters *A* and *B* belongs to sepiolite suspension with 6 wt% at 25 min, which is considered the optimized sample in the experiments applied. Furthermore, it can be observed that the extent of thixotropy embodied by the magnitude of *B* increases with increasing solid rate and homogenization time. This means that the high shearing effect is intensified by increasing solid rate and homogenization time, which could be explained in terms of a greater number of surface silanol groups created by increasing surface area.

4. Conclusions

The rheological properties of sepiolite suspensions homogenized in water by Ultra-Turrax high-speed rotor-stator homogenizer were investigated under various conditions in homogenization times, suspension solid concentration and distance between dispersing tool/bottom. The raw sepiolite of Turkish origin used in this study was quite heterogeneous with a moderate proportion (~53%) of the main phase. All the sepiolite suspensions homogenized Ultra-Turrax high-speed homogenizer exhibited non-Newtonian flow behavior that was characterized by a change in the viscosity with changes in the shearing rate and time. Moreover, the rheological properties of raw sepiolite were substantially affected by suspension solid rate and homogenization time variations. The higher the solid concentration the greater number of surface silanol groups on the surface of the sepiolite fibers and the increasing particle-particle interactions created by increasing the surface area that controls the rheological behavior of sepiolite suspensions. Assuming that the rheological behavior is drastically modified when >5 wt% and flow profile of 6 wt% homogenized sepiolite suspension is indicative of high thixotropy, whereas the flow behavior character of homogenized sepiolite suspension with the concentrations of 4 and 5 wt% can be seen to be moderately thixotropic with some yield stress. This is evident when the solid concentration of sepiolite suspensions is high, in which case homogenization time seems to affect the viscosity. This shows clearly that homogenization time has a pronounced effect on apparent viscosity, which is provided no longer than 25 min. Raw sepiolite's flow behavior and time-dependent/independent properties were analyzed and modeled by Power-law and Weltman models. The relatively good R^2 values (≥ 0.90) obtained for all sepiolite suspensions considering the downward curves showed that all sepiolite suspensions behaved like a non-Newtonian flow and expressed a shear-thinning behavior (pseudoplastic). Furthermore, shear stress and apparent viscosity decreased with the increasing shearing time, which also confirmed thixotropic behavior. Also, the Weltman model was appropriate to fit the time-dependency behavior of sepiolite suspensions. The most obtained value for model parameters *A* and *B* has belonged to sepiolite suspension with 6 wt% at 25 min, which was considered the optimized sample in the applied experiments. The final viscosity level increased from 0.82 to 12.55 Pa.s. Consequently, the rheological properties of sepiolite suspensions can be improved economically by Ultra-Turrax high-speed homogenizer as an enhancement method to improve the degree of dispersion of the sepiolite fiber bundles in water and hence increase viscosity and thixotropic behavior, which is an essential characteristic for industrial applications. Based on the data obtained, shearing time should be limited to 25 min, also in terms of a feasible and economical process.

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