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SYNTHETIC OIL GELS WITH ORGANOCLAYS IN THE FORMULATION OF MAGNETORHEOLOGICAL FLUIDS

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Abstract. Magnetorheological fluids (MRF) are smart composite materials that under an external magnetic field show a reversible solid-liquid transition in less than 10 ms. The purpose of this study was to evaluate which organoclays would jellify a synthetic oil for the formulation of MRF. Furthermore, seven dispersant additives for carbonyl iron powder were also evaluated. Fifteen different gelling additives from four different families: bentonites, hectorites, montmorillonites, and mixed mineral thixotropes (MMT) were dispersed in oil only, keeping the same concentration, without iron. The gels were tested through amplitude sweep and frequency sweep in oscillatory rheometry, to evaluate their viscoelastic behavior. The thixotropy of the gels was measured with the "three-interval" test in a rheometer. After selecting the best gelling additive to prepare MRF, seven dispersing additives were evaluated for magnetorheological effect and redispersibility after one year of sample preparation.

In the linear viscoelastic region, all MMT clays result in a weakly viscoelastic gel (G' ~ 100 to 300 Pa and G" ~ 30 to 50 Pa). Some of the bentonite clays jellified and others did not. The best organoclays were montmorillonites and hectorites, which formed consistent viscoelastic gels (G' ~ 1 to 5 kPa and G" ~ 70 to 250 Pa).

The best organoclay presented yield stress σ_0 = (42.3 ± 2.6) Pa, storage modulus G' = (2690 ± 201) Pa, and cohesive energy density (CED) = 98 mJ/m³, and it was selected to explore the rheology of MRF with seven ('A' – 'G') dispersant additives. All the MRFs were prepared with carbonyl iron powder HS (BASF SE) in oil gel with the same organoclay. All seven dispersant additives showed thixotropic recovery above 100% in the three-interval test.

Regarding the redispersibility test after one year, the MRF formulations A, B, C, and F resulted similarly, with the normal force up to 0.6 N. Additives D, E, and G, however, resulted in the hard-cake formation, being reproved. The formulation C normal force peak was FN = 0.39 N and Work = 9.4 mJ. We conclude that the MRF with dispersant C showed a better balance between thixotropy, MR effect, and, above all, good redispersibility.

Key words: Magnetorheological fluid; Organomodified clay; Redispersibility; Cohesive energy.

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1 INTRODUCTION

Although the term “composite” originated from the combination of polymeric resins with reinforcing fibers [1], magnetorheological (MR) materials (whether MR fluids, MR Gels, or MR Elastomers, etc.) are also composite materials [2]. In the case of magnetorheological fluids (MRF), the matrix is the continuous liquid phase, and the solid magnetic particles, the dispersed phase [3]. For magnetorheological elastomers (MRE), the continuous matrix is some elastomer, such as silicone rubber, for example [4]. As for magnetorheological gels (MRG), it can either be a gel formed by some copolymer dissolved in oil [5], or a gel of inorganic material, such as clays (natural, if the liquid phase is aqueous, or chemically modified, if the medium is a solvent or a synthetic oil, derived from hydrocarbons), for example [6].

In the case of MRF, the simplest formulations consist only of the dispersed magnetic phase and a continuous liquid medium. The most commonly used material during preparation is carbonyl iron powder (CIP). This material has a purity above 97% and consists of zero-valence metallic iron. As a continuous phase, the most common liquids used to prepare MRF are water or aqueous solutions, oils (mineral, silicone, synthetic, vegetable, etc.), perfluoropolyethers, glycols, ionic liquids, among a myriad of other MRF formulations [7]. Although some exotic liquid metals such as Gallium, Mercury, and Galinstan have been reported in the literature, these materials can form metallic alloys with the CIP, resulting in amalgams [8]. They are, therefore, not exactly MRF. Water-based MRF are commonly applied in MR polishing, while in the automotive industry, MR devices (shock absorbers, brakes, clutches, etc.) usually have oils (generally polyalphaolefin oil or silicone oil) as the liquid phase [7].

While physicists usually avoid additives that would complicate the analysis of the experimental findings (and the comparison with the theoretical predictions as well), engineers and scientists who design MR devices (and those interested in preparing MRFs), cannot avoid formulating MRFs with additives. In this context, the most used additives are dispersants, gelling agents, lubricants, anti-wear, anti-corrosives, anti-foams, etc [7].

An inherent and inevitable problem for anyone who has ever formulated an MRF is the sedimentation of the dense iron particles in a liquid medium with a much lower density. Another relevant problem, although this one is easier to solve, is the coagulation/aggregation of the particles caused by the van der Waals forces and, even more, the residual remanence of the CIP particles [9]. In this case, correctly selected dispersant additives can solve the problem. As for the former problem, the best one can achieve is to delay sedimentation and ensure an easy redispersion. Completely preventing sedimentation is impossible, however. Some might argue that with the advent of MRF's "younger brothers", such as MRE, MR Gels, or MR Foams, the sedimentation problem has been solved and eliminated. Nevertheless, there is a significant reduction in the range of control of these materials when compared to conventional MR fluids.

Among the most common gelling additives cited in patents and in the literature on MRF, fumed silicas and clays (natural or organomodified) stand out [10, 11]. This work aimed to investigate, among four main classes of clays, which one is the most suitable, out of 15 evaluated clays, to gel a synthetic polyalphaolefin (PAO) oil. These classes were: montmorillonite, bentonite, hectorite, and mixed mineral thixotrope (MMT) [12]. In a subsequent step, we chose a single clay (the one that was most effective in gelling the PAO oil, as measured by a higher G' value) and evaluated, among seven dispersant additives (fatty alcohols, fatty amines and commercial dispersants) which showed the best performance. The following responses were evaluated: a) the MR effect under magnetic field (on-state), b) the thixotropic recovery without magnetic field (off-state), c) the redispersibility of the samples under normal gravity, at rest and without applied field, in the periods of 3, 7, 30 and 365 days.

2 EXPERIMENTAL

2.1 Preparation of the thixotropic gels

To prepare the gels, fifteen types of clay, organized in four groups, were dispersed in polyalphaolefin oil (Durasyn PAO 162, INEOS), totaling 5 wt.% of the oil's mass. The gels were then homogenized with an IKA Ultra Turrax T-18 mechanical disperser at 6000 rpm for 30 seconds, followed by the addition of the chemical activator: propylene carbonate - water 95:5, and 33.3 wt.% of the clay's mass [13]. The mixture was homogenized again at 20,000 rpm for a minute, and their rheometry was investigated.

2.2 Rheometry of the gels

The samples that were jellified were taken to a rheometer (Anton Paar Physica MCR 301) for an oscillatory sweep test to measure the yield stress σ_0 and the storage modulus G'. The sample with greater values of G' and σ_0 was selected as an additive to prepare the MRF's. An oscillatory frequency sweep was also performed to analyze the complex viscosity η^* and to compare the storage and loss moduli (G' and G'').

2.3 Preparation of the magnetorheological fluids

After carefully selecting the best additive, seven MRF's were prepared with 50 vol% of carbonyl iron particles (d = 7.86 $g.cm^{-3}$), 49 vol% of carrier liquid (PAO + activator + dispersant), and 1 vol% of clay. A different type of dispersant was used in each preparation, and they were labeled A-G. Firstly, the polyalphaolefin oil was weighted, and the dispersant was added (0.8 wt% of the carbonyl iron particles). Then, the iron particles were added, and the mixture was homogenized with an IKA Ultra Turrax T-18 mechanical disperser at 6000 rpm for 2-3 minutes. The clay was added, the mixture was homogenized again, and the activator was added [13]. Finally, the sample was stirred vigorously at 20,000 rpm for 60 seconds to jellify the clays, and their rheology was studied.

2.4 Magnetorheology

After preparation of the MRF's, the following tests were performed:

2.4.1 Amplitude sweep (off-state): To obtain the linear visco-elastic range, the prepared MRF were subjected to amplitude sweeps in the absence of an applied magnetic field, at 25 ºC. A cone-and-plate configuration was used (diameter of 25 mm) with a gap d = 0.056 mm and frequency ω = 10 rad/s. The strain γ ranged from 0.001% to 100% [14].

2.4.2 Frequency sweep (off-state): To analyze the complex viscosity and to obtain the values of

G’ and G’’, frequency sweeps were carried out in the absence of an applied magnetic field, at 25 ºC. A cone-and-plate configuration was used (diameter of 25 mm), with a gap d = 0.5 mm and constant strain $\gamma = 0.05\%$ (linear viscoelastic range). The angular frequency ω was swept in decreasing order, from 500 rad/s to 0.05 rad/s [15].

2.4.3 Amplitude sweep (on-state): The MRF were also subjected to amplitude sweeps under an applied magnetic field, at 25 ºC. A cone-and-plate configuration was used (diameter of 20 mm), with a gap d = 0.5 mm and frequency $\omega = 10$ rad/s. The strain γ ranged from 0.001% to 100%, and three conditions of current and magnetic flux density were analyzed: 1) $I_1 = 0$ A, then $B_1 = 0$ mT, 2) $I_2 = 1$ A, so $B_2 = 192$ mT, and 3) $I_3 = 2$ A, with $B_3 = 398$ mT [14].

2.4.4 Magneto sweep: The MRF were then subjected to a mag sweep and the magnitude of the applied magnetic field was measured with a gaussmeter, at 25 ºC. A cone-and-plate configuration was used (diameter of 20 mm), with a gap d = 0.5 mm and constant frequency $\omega = 10$ rad/s. The strain γ ranged from 0.01% to 100%, and the current linearly ramped from 0 – 3 A [16].

2.4.5 Thixotropy: The thixotropy of each MRF was analyzed without a magnetic field (off-state), with a parallel plate configuration (20 mm of diameter) and gap d = 1 mm. The test was carried out with the so called “step test with three intervals” was employed: in the first and third intervals, the sample was sheared at a shear rate $\dot{\gamma} = 0.01$ s^{-1}. In the second interval, the sample was sheared at $\dot{\gamma} = 100$ s^{-1}. The shear viscosity was followed along all the time. To the reader interested in more detail on this assay for measuring the thixotropy of rheological materials, the Mezger reference is recommended [17].

2.4.6 Redispersibility: Finally, each sample had their redispersibility measured with the rheometer, after sedimentation at rest and under normal gravity. The normal force and the work done were then computed. The tests were repeated after three days, one week, one month and one year [18].

3 RESULTS AND DISCUSSION

3.1 Organoclay gels and amplitude sweep

Figure 1 shows the storage modulus as a function of the strain amplitude for four OM clays: #6 is a smectite (or montmorillonite), #9 is MMT, #13 is a hectorite, and #15, a bentonite. From Figure 1, one can see that the MMT generates the weakest gel, with only G’ ≈ 200 Pa in the linear viscoelastic (LVE) region, while the other clays gave stronger gels, increasing in the following order: bentonite (G’ ≈ 750 Pa) < hectorite (G’ ≈ 2,800 Pa) < smectite (G’ ≈ 4,600 Pa). With these results, the best OM clay #6 (smectite) was chosen, and all MRF were prepared with this clay.

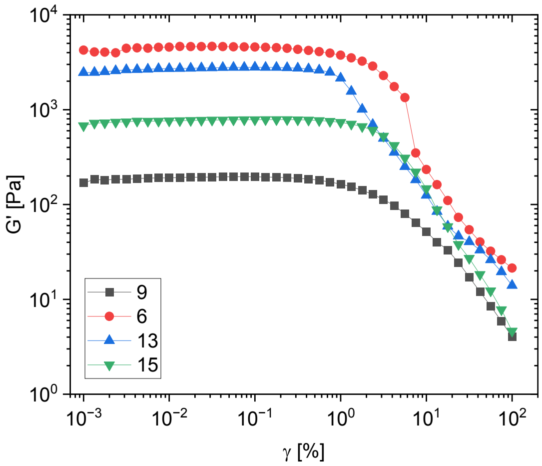

Figure 1: The storage modulus G' as a function of the amplitude strain γ for the gels obtained with OM clays and PAO oil only, at 25 °C.

According to Ross-Murphy, if the complex viscoelastic modulus G* is divided by the $G^*_{\gamma\to0}$ (i.e. the G* value for strain γ tending to zero), and a graph of this ratio is constructed as a function of the strain γ (%), one can classify the material in a 'weak gel' if the ratio $G^*/G^*_{\gamma\to0}$ drops almost to zero, for strain < 1%. If this ratio remains constant around one, for strain values up to ~100%, the material can be considered a strong gel [19]. In this case, we have that the gels formed with the different clays resulted in weak gels to moderate gels. On the other hand, according to Mezger, whenever G' > G" in an oscillatory amplitude sweep, the value of G' can be considered as a measure of the "gel strength" [20]. Therefore, gelling agents based on smectite (#6), and hectorite (#13) clays resulted in stronger gels than bentonite (#15) or MMT (#9) clays.

3.2 Organoclay gels and frequency sweep

Figure 2 shows the viscoelastic moduli as a function of the frequency sweep (under constant amplitude strain in the LVE region) to verify their behavior: a strong gel, a weak gel, or a viscoelastic liquid. One can see that the storage modulus G' was around 2 kPa, while the loss modulus G" was around 200 Pa in the entire angular frequency range. The complex viscosity η* continuously decreases with increasing frequency, as is typical in materials with shear thinning behavior. Besides, the complex viscosity has a constant slope approaching −1, which is also typical of a gel. According to Steffe [21] and also Almdal *et al* [22], a material in a 'gel' state, when analyzed by oscillatory rheometry, must present two main characteristics: a) the value of G' greater than G" in several orders of frequency and b) constant G', virtually invariable with frequency. Therefore, as G' ≈ ten times greater than G", and the two modules are relatively constant and independent of frequency, corroborates the amplitude sweep analysis, as this is also characteristic of the rheological behavior of gels.

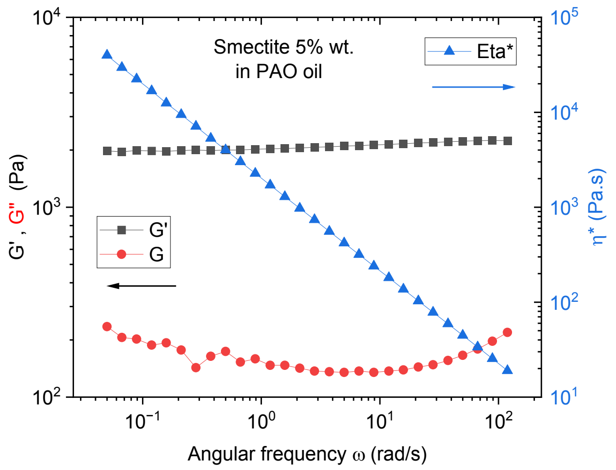

Figure 2: Viscoelastic moduli G’ and G” (left axis) and complex viscosity η* (right axis) as a function of the angular frequency ω for the strongest gel obtained with OM smectites (5 wt%) in PAO oil only, at 25 °C.

3.3 The yield stress of the organoclay gels, by oscillatory amplitude sweep

Figure 3 shows the storage modulus as a function of the shear stress for gel #6. In this type of plot, the value where the storage modulus G’ decreases abruptly corresponds to the yield stress σ itself. The measurements were performed in triplicate, with previously untested samples.

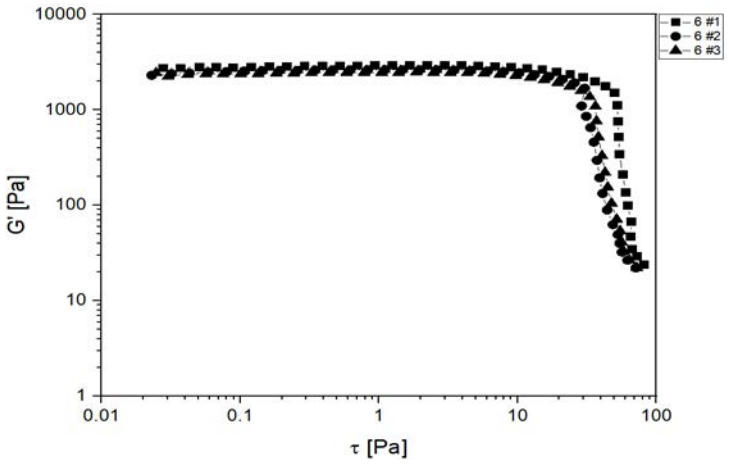

Figure 3: The storage modulus as a function of the shear stress for the best gel formulation, an OM smectite (5 wt%) in PAO oil only, at 25 °C.

In figure 3, it can be seen that the average value of the yield stress, associated with the shear stress value (abscissa), in which the storage modulus G' has a sharp drop, is around 40 Pa.

3.4 The amplitude sweep and the cohesive energy density

With the oscillatory amplitude sweep curves, it is also possible to compute an important parameter for the gel, the so-called cohesive energy density (CED). It is calculated with the help of Equation 1:

$$C.E.D. = \frac{1}{2}\, G'(\gamma_{cr})^2 \qquad (1)$$

where G' is the storage modulus value at its plateau region (LVE range) and γ_{cr} is the critical strain (without units and not in percentage), that is, the maximum value of strain before that the storage modulus G' begins to decrease [23]. The figure 4 shows the procedure used to compute the cohesive energy density of gel #6:

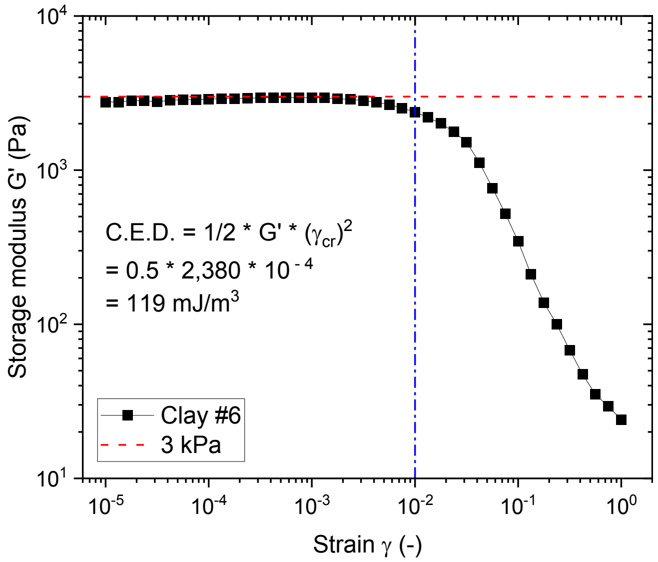

Figure 4: The storage modulus as a function of strain for the best gel formulation, an OM smectite (5 wt%) in PAO oil only, at 25 °C. Although the dashed lines are arbitrary, the region where there is a 20% reduction of the maximum value of G' can be taken as the critical strain. In this case, G' = 2,380 Pa, γ_{cr} = 0.01 (no units) and, therefore, the Cohesive Energy Density ≈ 120 mJ/m^3.

The cohesive energy density is a useful parameter to evaluate the strength of inorganic and organic gels, as it measures the mechanical energy required to break weak interactions. The greater its value, the more resistant a material is to flow and slower must be the sedimentation of iron micro particles through the gel [23].

3.5 Amplitude sweep (on-state):

After choosing clay #6 as an additive, the MRF's were prepared, and their rheology was investigated. Firstly, the yield stress values σ of each fluid were computed from a Storage Modulus versus Shear Stress plot, as shown in Figure 5.

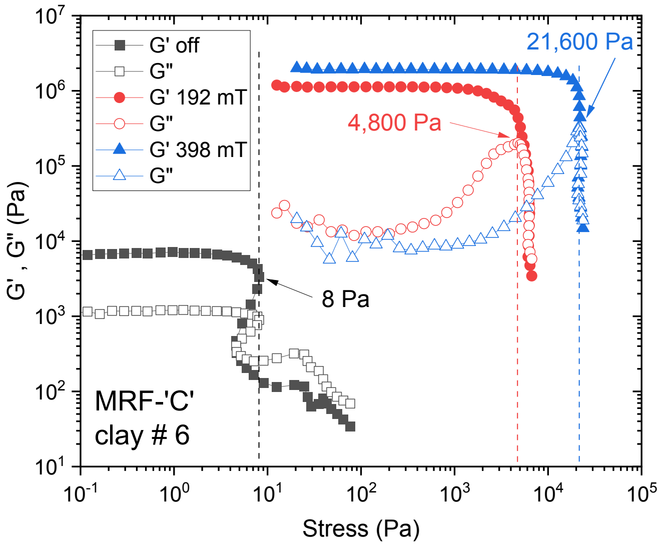

Figure 5: Amplitude sweep test for MRF with clay #6 and dispersant 'C', without magnetic field (black squares), and under B = 192 mT (red circles), or B = 398 mT (blue triangles). The dashed lines indicate the yield values taken for the calculation of the relative MR effect.

The relative magnetorheological effect of each fluid was calculated, according to Equation 2:

$$MR_{relative} = \frac{(\sigma_B - \sigma_{off})}{\sigma_{off}} \tag{2}$$

where σ_B is the yield stress value on the on-state, and σ_{off} is the yield stress on the off-state. As an example of the calculation, taking the yield stress values from figure 5, where σ_{blue} = 21600 Pa (B= 398 mT) and σ_{black} = 8 Pa (B = 0, off state), results in a relative MR effect value of 2700.

3.6 Thixotropy

As for the thixotropic response, all MRF formulations showed fast (in less than 2 seconds) and complete (above 100%) thixotropic recovery. Therefore, we chose not to present the corresponding graph, because qualitatively, all additives were satisfactory in terms of this property, which also demonstrates that it depends almost exclusively on the chosen clay.

3.7 Redispersibility

Finally, the redispersibility of each fluid was analyzed, and the results are shown in Figure 6. For brevity and to avoid repeating results that are qualitatively unimportant, we will only present the redispersibility results after one year.

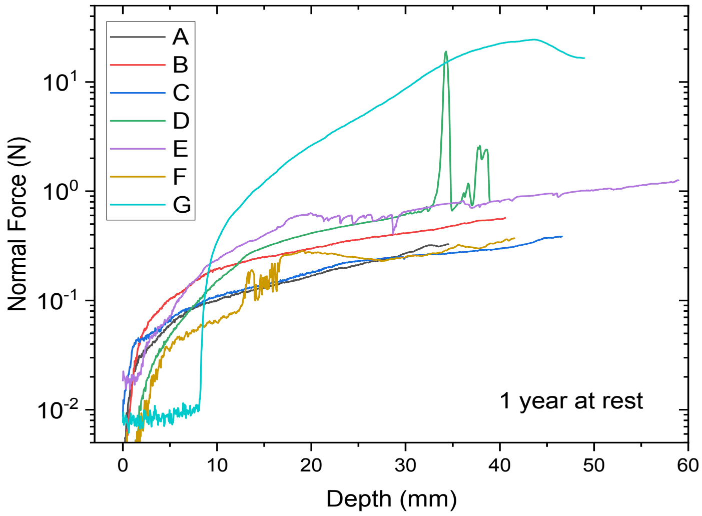

Figure 6: The redispersibility test after one year at rest, under normal gravity. The normal force is shown as a function of the penetration depth of the vane tool for all the MRF formulations. The amounts of OM clay were kept the same but employing seven different dispersant additives.

The rheometry results for each magnetorheological fluid are summarized in Table 1:

Table 1: Example of the construction of one table

Sample	MR Effect	G’$_{off}$ (Pa)	Force (N)	Work (mJ)
A	1000	30000	0.33	3.4
B	400	50000	0.57	12.0
C	2700	7000	0.39	9.4
D	10000	5000	19.00	27.6
E	130	60000	1.27	38.4
F	130	90000	7.61	7.6
G	10000	1000	24.40	414.5

From Table 1, it is possible to draw some conclusions. There seems to be an inverse correlation between the storage modulus values (off-state) and the redispersibility results: very low values of G' led to the formation of hard sediments after one year. This can be noticed for the formulations prepared with additives D and G.

As the relative MR effect depends on the rheological properties in the absence of field, minimizing the G' values in off-state would be the ideal. However, it is not enough to focus only on a stronger MR effect without also ensuring easy redispersibility after sedimentation. After all, if the MRF becomes a difficult material to redisperse, it becomes useless. Note that the MR effect was greater for the samples with the same additives, D and G: they were the best in terms of the MR effect, but they were also the worst in terms of redispersibility.

Therefore, of the seven additives tested, those that gave a good MR effect without seriously compromising redispersibility were additives A and C, with a slight advantage for the latter regarding the MR effect.

4 CONCLUSIONS

Among the 15 organomodified (OM) clays evaluated in this work, only three of them (the MMT clays) did not jellify when mixed with polyalphaolefin oil. Among the remaining 12 that jellified, the rheometry results indicated clays #5, 6 and 14 (smectites, hectorites and bentonites) were good candidates as thixotropic additives for the preparation of MR fluids. Clay #6 was chosen as an additive for presenting: the highest value of yield stress $\sigma_0 = (42.3 \pm 2.6)$ Pa; the largest storage modulus $G' = (2690 \pm 201)$ Pa; and the highest CED value = 98 mJ/m³. In addition, the complex viscosity of the clay used in the MR formulations increases with wt%, as would be expected.

Using clay #6, it was possible to prepare seven stable and functional MRF's by dispersing HS carbonyl iron particles in a matrix of PAO oil, all with the composition of 90 wt.% iron, 51 vol% in solids (iron + clay), and 1 vol% in clay.

The amplitude sweep test with the application of a magnetic field allowed us to verify the abrupt change of the rheological properties for all fluids A-G. The highest values of the MR effect (10000x) were verified for fluids D and G.

The three interval thixotropy tests showed that all MR fluids in this study have a thixotropic behavior with viscosity recovery above 100% for all additives. In addition, the recovery was very fast, reaching 100% in less than 2 seconds, immediately after the shear reduction step in the third interval.

The redispersibility tests showed that for the MR fluids D and G, there was a great deterioration in the stability of the suspensions after one year of preparation. This is evidenced by the high values of normal force and mechanical work recorded in the redispersibility test: $F_N = 19.00$ N and $W = 27.6$ mJ for fluid D and $F_N = 24.40$ N and $W = 414.5$ mJ for fluid G. On the other hand, formulations A and C showed excellent redispersibility results; even after one year of preparation, the normal force results were less than 0.6 N. For fluid C especially, the normal force and work values after one year were 0.39 N and 9.4 mJ, respectively.

The results of all the tests allowed us to conclude that the MR fluid prepared with dispersant C showed a better balance between the measured properties: G'_{off}, σ_0, MR effect, and good redispersibility after one year of preparation. The use of dispersant C and the thixotropic gel #6 resulted in a functional and stable MR fluid with at least one year of shelf life.

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