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Magneto-rheological fluids redispersibility - a factorial design study of phosphate shell on carbonyl iron powder with dispersing additives

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Abstract. We showed, in a previous paper, that Magneto-Rheological Fluids (MRFs) have different rheology when prepared with Carbonyl Iron Powders (CIP) phosphate (coated or uncoated). This was especially so when done without a magnetic field. This paper employs factorial design to examine the redispersibility and rheology of some MRF formulations; we use the same CIPs but with different dispersing additives. The factors are: CIP A (uncoated) or B (phosphate shell); additives with carboxylic acid or primary amine as the polar group; and n-octyl (C_8H_{17}) or n-dodecyl ($C_{12}H_{25}$) as the alkyl hydrocarbon chain (R-). CIP B was much more redispersible than CIP A, especially with amine additives; typical work values were < 5mJ @ 20 mm depth. In terms of viscosity, CIP A generated lower values, at shear rates above 100 s^{-1}. It also realized higher yield stress values (H_o = 300 kA/m) than CIP B (50% and beyond).

1. Introduction

As shown in a previous paper [1], Magneto-Rheological Fluids (MRFs) prepared with phosphate coated or uncoated carbonyl iron powders (CIP) have different rheology. This happens because Hydrophilic-fumed silica interacts with the phosphate (via hydrogen bonding) and it is especially true when a magnetic field is absent. The challenge in MRF formulation [2, 3] is redispersibility. This quality was measured, according to the test described by *Kieburg et al* [4]. In this test the work is measured by how far a steel blade, penetrates vertically downward into a test tube, at a constant speed. Each test tube contained 10 mL of an MRF formulation that was centrifuged for 15 minutes at 2000 'g'. This paper makes use of factorial design of experiments [5, 6] to study the redispersibility of some MRF formulations; the formulations include two carbonyl iron powders with different dispersing additives. The work (mJ) comprises the response variable for the factorial design; it was measured with a normal force cell built-in rheometer (Anton Paar – Physica MCR-301).

2. Experimental

The MRF formulations were made with a food grade, poly(alpha-olefin) oil. In addition to this oil (balance) the formulations consisted of the dispersing additive (0.8% w/w) and the CIP (80% w/w). A modified montmorillonite clay (0.3% w/w) was used as thixotropic agent. In each MRF formulation a high shear homogenizer (Ika – Turrax T-18) dispersed the CIP. The MRF samples were prepared in

duplicate and measured twice. More details about the CIP can be found in reference [1]. All the rheological measurements were obtained with a stress controlled rheometer Physica MCR-301.
Thus, the factorial design is 2^3 (2 levels, 3 factors). Table 1 displays the full cube of possible combinations, with duplicate samples, and randomized order.

3. Results and Discussion

Table 1. MRF 2^3 factorial design and yield stress values

Std Order	CIP	Polar Group	Alkyl	Yield 1	Yield 2	Yield 3	Yield 4	Yield mean
9	A	NH_2	Octyl	17.0	15.9	15.9	-	16.3
16	B	COOH	Dodecyl	7.1	7.6	7.1	9.1	7.7
11	A	NH_2	Dodecyl	16.8	18.0	17.4	-	17.4
4	B	NH_2	Dodecyl	10.5	10.0	10.9	11.2	10.7
2	B	NH_2	Octyl	11.2	12.2	-	-	11.7
15	A	COOH	Dodecyl	17.6	18.1	17.1	-	17.6
7	A	COOH	Dodecyl	18.1	18.5	18.1	-	18.2
1	A	NH_2	Octyl	18.5	16.0	17.5	18.4	17.6
14	B	COOH	Octyl	11.6	10.5	9.4	11.2	10.7
5	A	COOH	Octyl	15.4	13.6	14.0	-	14.3
12	B	NH_2	Dodecyl	10.3	9.8	9.7	9.0	9.7
3	A	NH_2	Dodecyl	18.5	18.2	17.1	-	17.9
6	B	COOH	Octyl	13.5	10.2	11.3	11.6	11.7
10	B	NH_2	Octyl	9.8	10.2	9.7	-	9.9
13	A	COOH	Octyl	14.0	14.2	14.0	-	14.1
8	B	COOH	Dodecyl	7.8	7.7	8.1	-	7.9

Figures 1 and 2 show the redispersibility as curves of force versus depth, for carbonyl iron powders A (without phosphate) and B (with phosphate shell). One sees first that Powder B, with its smooth force curves and absence of peaks, generally did better than A. With the exception of the octanoic acid, Powder B was nearly additive-independent. The second thing one sees is that with Powder B amines outperformed acids while with Powder A acids outperformed amines.

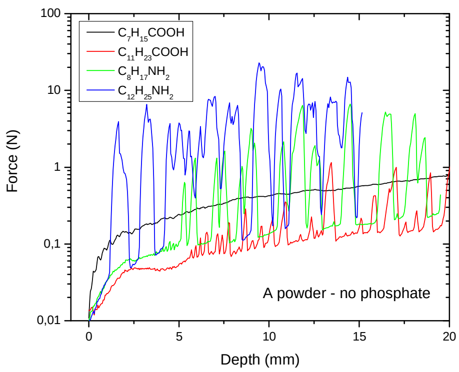

Figure 1. Force as function of blade depth, for MRF prepared with CIP A.

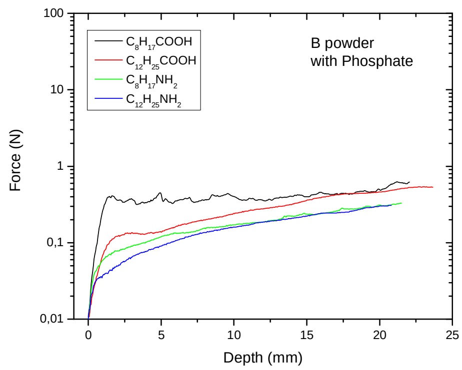

Figure 2. Force as function of blade depth, for MRF prepared with CIP B.

(Dispersing additives: 1% w/w of Octanoic Acid - Black; Dodecanoic Acid – Red; n-Octylamine - Green; or n-Dodecylamine – Blue.)

Figure 3 shows the viscosity curves (no field) for the 8 MRFs. Powder B generated MRFs with higher viscosities, above 100 s^{-1}. Figure 4 shows the shear stress versus shear rate for the 8 MRFs, under applied field H_o = 300 kA/m. It is evident that Powder A was better than B since the yield stress values of MRFs with Powder A were higher than with Powder B. Curiously, the additives seem to have an effect, though secondary, on the yield value.

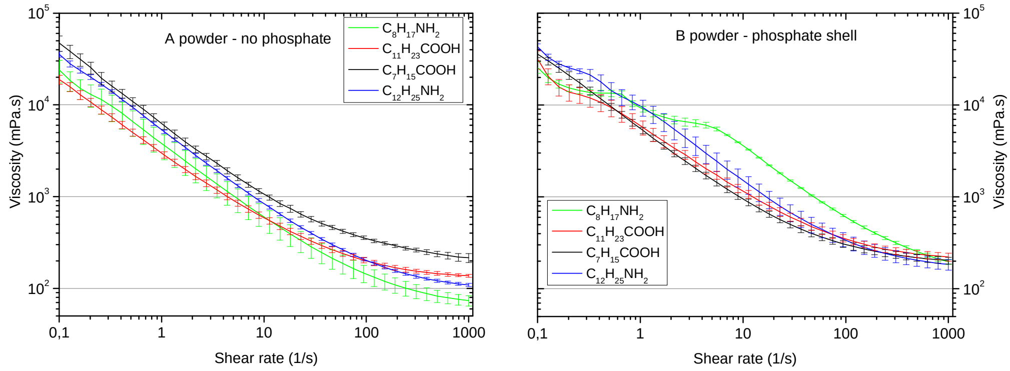

Figure 3. Viscosity curves for the MRF with CIP A (left) and B (right) without magnetic field (T = 25°C. Each point is the mean of the sample duplicate and two measurements with an SD bar. Same color legend as Figures 1 & 2.)

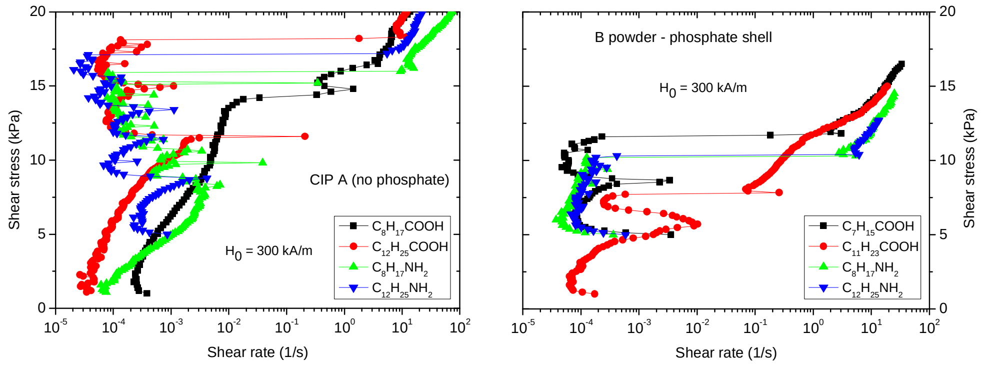

Figure 4. Flow curves for MRF prepared with CIP A (left) and B (right), measuring yield stress. (Applied field strength H_o = 300 kA/m, measured as an empty gap. Colors as before.)

Table 2 resumes the terms, coefficients, standard error, T and P-values of the factorial analysis. Boldface emphasizes relevant factors and interactions. Figure 5 shows the main effects, interactions, cube plots, and the Pareto chart for the yield stress response. Powder type, the evidence reveals, produces the main effect; phosphate coating reduces the yield stress. Polar group reveals an important effect and the HC chain length's effect is irrelevant.

Table 2. Estimated effects and coefficients for yield stress mean (coded units)

Term	Effect	Coef	SE Coef	T	P
Constant		13.331	0.1728	77.13	**0.000**
Powder Type	- 6.696	- 3.348	0.1728	- 19.37	**0.000**
HC Chain Length	0.115	0.057	0.1728	0.33	0.749
Polar Group	1.125	0.563	0.1728	3.25	**0.012**
Powder Type*HC Chain	- 2.110	- 1.055	0.1728	- 6.11	**0.000**
Powder Type*Polar Group	- 0.117	- 0.058	0.1728	- 0.34	0.744
HC Chain*Polar Group	- 0.060	- 0.030	0.1728	- 0.17	0.866
Powder Type*HC Chain*Polar Group	1.431	0.716	0.1728	4.14	**0.003**

S = 0.69 R^2 = 98.22% R^2 (adj) = 96.66%

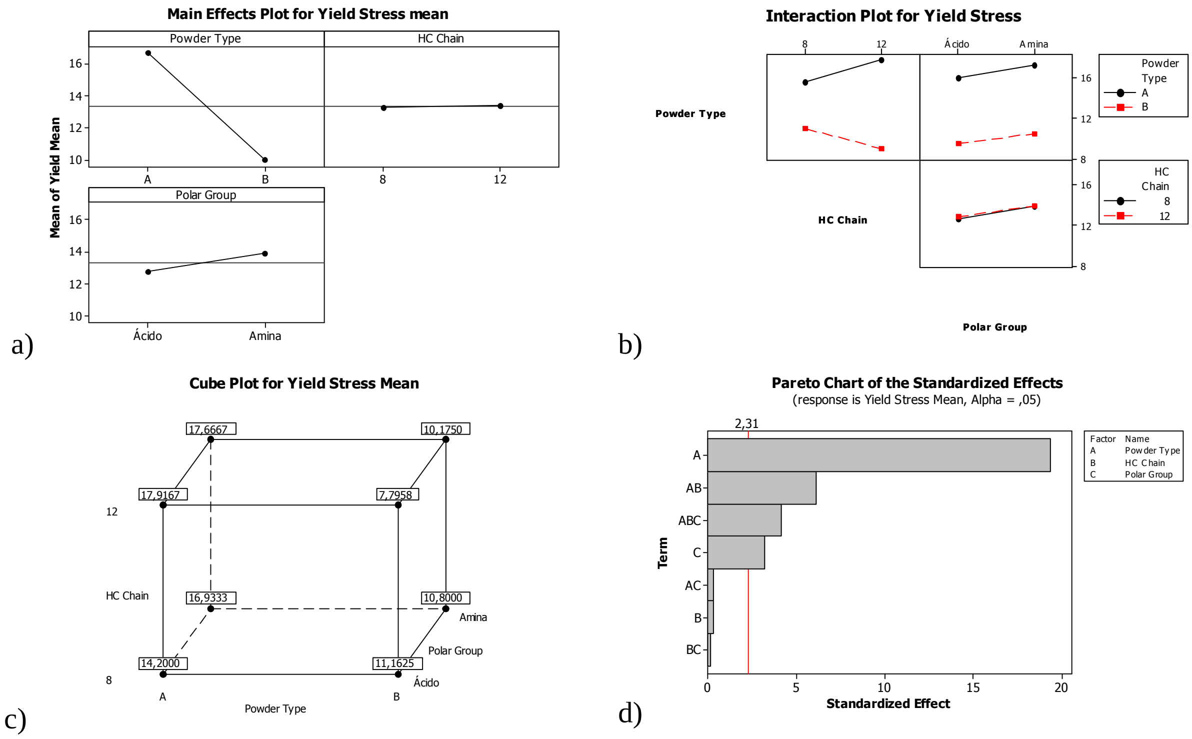

Figure 5. Factorial plots for yield stress mean analysis

a) Main effects plot; b) Interaction Plot; c) Cube Plot; d) Pareto Chart.

4. Conclusions

Carbonyl Iron Powder B (with phosphate shell) was much more redispersible than Powder A (without phosphate); this was especially true with amine additives. Powder A brought about lower viscosities, at shear rates above 100 s^{-1}. Powder A also generated higher yield stress values (H_o = 300 kA/m), at a rate of 50% and higher. It seems that additives also affected MRF behavior in magnetic fields. In the future, this aspect will be investigated using oscillatory tests.

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