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

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As shown in a previous paper [1], Magneto-Rheological Fluids (MRFs) prepared with phosphate coated or uncoated carbonyl iron powders (CIP) have different rheology, due to the presence of interactions between hydrophilic fumed silica and the phosphate, through hydrogen bonding. This especially happens without magnetic field.

Redispersibility is a challenge in MRF formulation [2,3] and it was measured through the work to vertically penetrate a steel blade at constant speed, into a test tube with 10 mL of each MRF formulation after centrifuging it @ 2000 'g' for 15 minutes, according to the test described by *Kieburg et al* [4].

The MRF formulations were made with a poly(alpha-olefine) oil, food grade. Besides the oil (balance); 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. A high shear homogenizer (Ika – Turrax T-18) was used to disperse the CIP in each MRF formulation. The MRF samples were prepared in duplicate, and the measurements were twice repeated.

In this paper, factorial design of experiments was applied to study the redispersibility of some MRF formulations of two carbonyl iron powders with different dispersing additives [5,6]. The response variable for the factorial design was the work (mJ) measured with the normal force cell built-in a rheometer (Anton Paar – Physica MCR-301).

Figure 1 shows the results and settings of a 2-level factorial design for the factors of (i) carbonyl iron powders A or B (uncoated or phosphate shell); (ii) additives with carboxyl or primary amine as polar group and (iii) n-octyl (C_8H_{17}) or n-dodecyl ($C_{12}H_{25}$) as alkyl hydrocarbon chain (R-). The presence of several interactions – what makes the DOE approach a very suitable methodology – can be seen in the Pareto chart. The main effect plot shows the individual effect of the variables on the mean work resulting in a natural choice for the minimum work for Powder of B, HC Chain of 8 and Polar Group of Acid. The second order interactions are also significant and represented by the unparallel lines in the interaction plot. The cube plot shows the experimental space for the considered factors revealing the Work mean.

Table I shows the generalized regression model considering two scenarios of experimentation. The first one was obtained when the statistical model included a specific outlier in the sample. In the second model the outlier was removed showing an excellent curve fitting for a nonlinear model. Both scenarios have indicated the optimization direction of minimum work to redisperse the MRF by adopting the above mentioned levels. The power of the DOE methodology was fundamental to understand the complex interactions between the factors CIP and the additives.

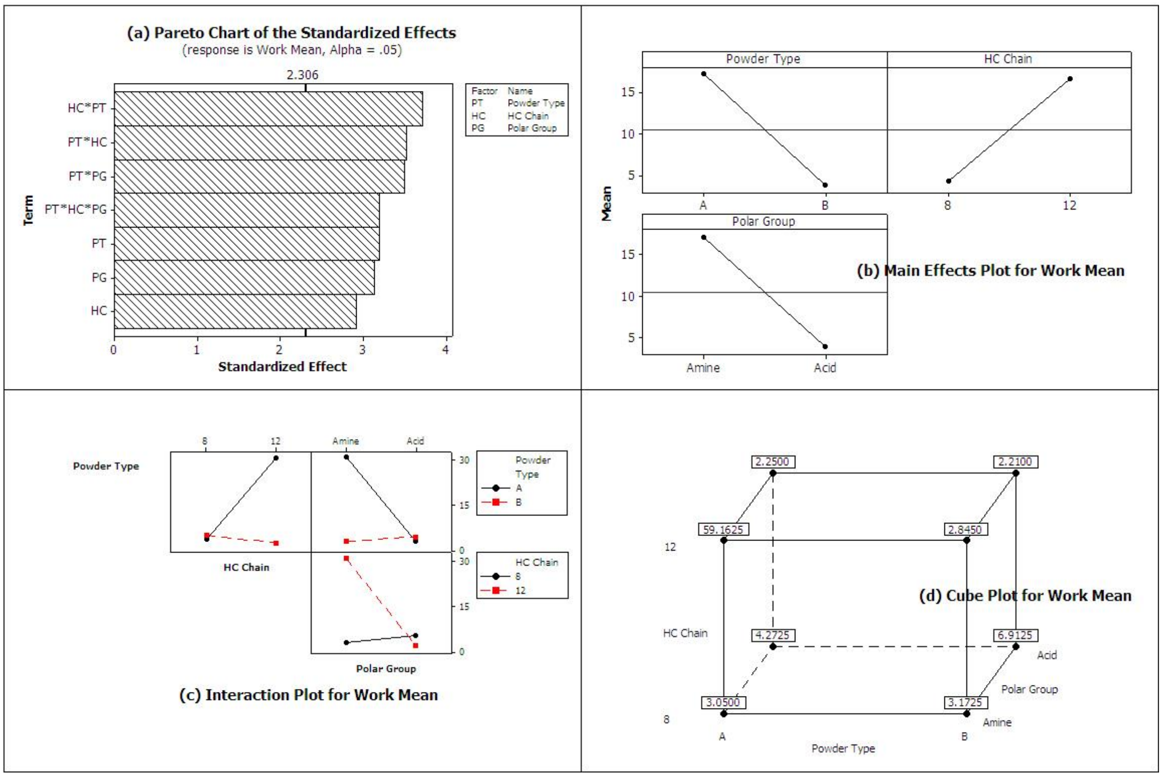

Fig. 1 Factorial plots for the experimental study.

Table I – Regression model for two experimental scenarios

With Outlier					
Term	**Effect**	**Coef**	**SE Coef**	**T**	**P**
Constant		10.484	2.096	5.00	0.001
Powder Type	-13.399	-6.699	2.096	-3.20	0.013
HC Chain	12.265	6.132	2.096	2.93	0.019
Polar Group	-13.146	-6.573	2.096	-3.14	0.014
Powder Type*HC Chain	-14.780	-7.390	2.096	-3.53	0.008
Powder Type*Polar Group	14.699	7.349	2.096	3.51	0.008
HC Chain*Polar Group	-15.627	-7.814	2.096	-3.73	0.006
Powder Type*HC Chain*Polar Group	13.440	6.720	2.096	3.21	0.013
S = 8.38537 PRESS = 2250.06					
R-Sq = 90.64% R-Sq(pred) = 62.57% R-Sq(adj) = 82.45%					
Removing Outlier					
Term	**Effect**	**Coef**	**SE Coef**	**T**	**P**
Constant		8,498	0,2052	41,40	0,000
Powder Type	-8,879	-4,439	0,2052	-21,63	0,000
HC Chain	7,745	3,872	0,2052	18,87	0,000
Polar Group	-8,351	-4,176	0,2052	-20,34	0,000
Powder Type*HC Chain	-10,806	-5,403	0,2052	-26,33	0,000
Powder Type*Polar Group	10,450	5,225	0,2052	25,46	0,000
HC Chain*Polar Group	-11,379	-5,689	0,2052	-27,72	0,000
Powder Type*HC Chain*Polar Group	8,645	4,322	0,2052	21,06	0,000
S = 0,820981 R-Sq = 99,79% R-Sq(adj) = 99,61%					

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