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Chemical And Rheological Properties Effects From Different Parameters In Horizontal Agitator Bead Mill Process On Sulfur Dispersion

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Abstract

Solid-liquid interactions are often associated with particle agglomerations and incomplete homogeneous system; thus grinding mill is one of the best unit operation employed by industries as a mean to counteract such drawbacks. Separately, knowing that the target of any processing plants is to reduce wastages by optimizing their unit operation's parameters, this paper detailed relatively on the approach to accomplish the organizational goal – by applying Taguchi's experimental design and analysis from Six Sigma. The optimization analysis has been successfully employed in this study; by the production of sulfur dispersion with its grinding mill's motor speed and pump rate as factors, whereby the dispersion's viscosity, pH, Total Solids Content (TSC) and particles size in D90 as their consequent responses. The experimental run was done in an industrial-scale horizontal agitator bead mill process with 60 liters of production capacity. Despite on the reduced number of experimental runs using Taguchi's L_9 orthogonal array, a total of 27 total runs have been performed and the optimized parameters can be seen at relatively higher motor speed and pump rate of 700 to 800 revolution per minute (rpm) and at 350 liters per minute (L/min) respectively; via the analysis of graphical representations on the responses of both factors towards their respective smaller-the-better means and larger-the-better S/N ratio methods. Plus, it can be seen that all of the sulfur dispersion's chemical specifications are affected by both the experimental factors, but most predominantly is the particle size. Since sulfur dispersion has been widely used in the incorporation from rubber compounding procedure, this study shall provide a way in continuous product quality improvement – especially in rubber or polymer business and industrial applications.

Keywords:grinding mill; optimization; sulfur dispersion; Taguchi.*

Introduction

Generally, a lot of industries in many areas such as chemicals, dry powders, ceramics, composite materials, mineral processing and others are demanding foreither fine or ultra-fine particles as feed material in their end product applications (Kotake *et al.*, 2011; Sabah *et al.*, 2013). Milling, grinding mill or comminution is a complex size-reduction mechanism that able to break down single crystal unit into smaller units with the aid of mechanical means. Precisely, milling process creates mechanical stress via repeated collisions ofparticles with balls or beads of particular size and material type (Tadros, 2017; Ranu *et al.*, 2015; Tangsathikulchai & Austin, 1989).

A lot of optimization studies have been done pertaining to grinding mill conditions which particularly focused on the milling parameter optimization for their targeted properties of the final products; of those include on particle size reduction of barium titanate nanocrystalline powders which conclude increasing milling time and speed result in gradual reduction in particle size until 18nm saturation value (Nath *et al.*, 2010); milling of quartz particle at both dry and wet milling conditions where the particle size distribution width was lowered by using smaller grinding balls in wet condition and larger grinding balls in dry condition (Sukanto *et al.*2019);optimization of ball milling process for silica sand tailing at 100 rpm milling speed, 15:1 ball-to-powder weight ratio (BPR) and 120 minutes milling time according to Particle Size Analyzer (PSA) (Sukanto *et al.*, 2019); on median particle size (D50) and specific surface area (SSA) of nanostructured tungsten carbide-cobalt, WC-Co powder which mentioned that except for weight ratio of milling balls to the powder material, parameters such as milling balls size, type of medium, volume of milling medium and rotation speed of planetary ball milling give significant SSA reduction (Zhang *et al.*, 2008); not to forget, study on silica sand nanoparticles production study using low speed ball milling method showed ball to powder weight ratio as the most influential milling parameter for the silica performance characteristics (Rizlan & Mamat, 2014).

Separately, the term “dispersion of solids” describes a physical mean whereby solid aggregates or particles are suspended as colloid and dispersed by the aid of surfactants and action of agitation in a liquid to obtain a highly uniform colloid or slurry (Atiemo-obeng *et al.*, 2004; Ottino *et al.*, 1999) – which in particular applied inclusively inindustries that deal with polymer such as rubber, that uses latex with other additives that are initially prepared as solid-liquid dispersions. One of these additives includes vulcanizing agent that uses powder (solid-form of) sulfur as its main raw material.Obviously a lot of studies have been done on different milling parameters over the products’ behavior but lesser study was done particularly on sulfur dispersion, as detailed in this paper. Sulfur particles need to be dispersed and milled in chemical plant at industrial scale especially for rubber applications, in their latex compounding procedure. It is equally important to study the effects of different process parameter towards the behavior of this vulcanizing agent in terms of its dispersion degree, which in the end might affect the properties of the end rubber-product (Brands *et al.*, 2001). One of the key indicators to measure the dispersion behavior is by observing its rheological properties or chemical characteristics. Figure 1 shows the illustration on process of dispersing solids in aqueous system such as sulfur dispersion.

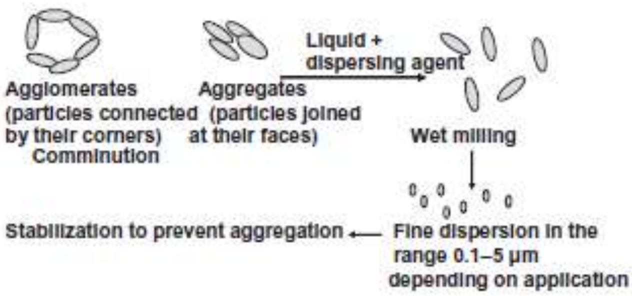

Figure 1: Dispersion of solids in aqueous system employing milling process (wet milling) (Tadros, 2017).

Experimental Details

In this project, the raw materials preparation include High-Pure Double-Refined sulfur powder grade supplied by Taiko Group of Companies, mixture of surfactants in a masterbatch from Behn Meyer Group of Companies, and deionized water. The mixing process of these materials in the mixing tank will follow the formulation prepared as in Table 1.

The process generally starts from the transportation of raw materials in Table 1 into an industrial scale of four metric tons (4 MT) mixing tank according to the sequence for mixing purpose with required dosage (by mass percentage) and left to stir for 30 minutes at 800 rpm, followed by grinding mill in a horizontal agitator bead mill machineof 60L mill capacity. Optimization of two factors – motor speed and pump rate for sulfur dispersion milling process – was done following Taguchi’s L_9 Orthogonal array as designed in Table 2 and Table 3 and the sample output is collectedafter 15minutestime.

Table 1: Ingredients in preparing sulfur dispersion

Ingredients	Mass percentage (%)
Water	34.0
Surfactant mixture (Master batch)	6.0
Sulfur Powder	60.0
TOTAL	100.0

Table 2: Control Factors and Variables (Levels) for Sulphur dispersion grinding mill process

Control Factors		Unit	Experimental Factor (Levels)		
			1	2	3
A	Motor Speed	Rpm	800	700	600
B	Pump Rate	L/min	750	550	350

Table 3: Taguchi's L_9 Orthogonal Array (for Sulphur dispersion grinding mill optimization)

Run No.	Columns on Experimental Factor	
	A Motor Speed	B Pump Rate
1	1	1
2	1	2
3	1	3
4	2	1
5	2	2
6	2	3
7	3	1
8	3	2
9	3	3

The sulfur dispersion samples were taken accordingly following Table 3 conditions set on the agitator bead mill machine. A small amount of the sulfur dispersion samples were taken and diluted with deionized water to be tested using HORIBA Particle Size Analyzer to obtain the value of D90 from the size distribution curve. Accordingly, the total solids content (TSC) of the sulfur dispersions were also measured and calculated as in Eq. 1. Plus, digital pH meter METTLER TOLEDO was also used that counts to 1 decimal place sensitivity, to measure the pH of each sulfur dispersion runs; and lastly fixed viscometer of Brookfield brand, RV-02 spindle and its stirring speed of 100 rpm will be used to observe the viscosity of the sulfur dispersions in centipoise (cP).

$$\text{TSC \%} = \left[\frac{\text{(weight of dry sample+container)-(weight of empty container)}}{\text{(weight wet sample+container)-(weight empty container)}}\right] \times 100\ \% \qquad \text{(Eq. 1)}$$

Results Analysis And Discussions

In this study, the sulfur dispersions were analysed using Six-sigma's Taguchi-based orthogonal array. Since the experiment was done in industrial scale, this method is the most suitable method to improve the product's quality and reduces time and cost for every industrial applications. Considering the availability and limitations in industrial scale, the experimental controllable factors have been chosen to be motor speed and pump rate of milling machine on the sulfur dispersions. For each parameter combination settings, sample was taken and three readings was done at different sulfur conditions: (i) freshly taken output, (ii) after 7 days stored at 25ºC room temperature, and (iii) after 22 hours stored at 70ºC oven temperature.

Table 4: Sulfur specifications result from first sample reading: freshly taken output.

Motor Speed (rpm)	Pump Rate (L/min)	Viscosity (cP)	pH	TSC (%)	D90 (μm)
800	750	472	9.5	61.31	7.571
800	550	610	9.5	62.99	6.840
800	350	598	9.6	61.63	6.183
700	750	470	9.7	62.05	8.558
700	550	646	9.9	61.84	7.660
700	350	616	9.5	62.31	6.902
600	750	370	9.7	62.43	10.066
600	550	432	9.7	62.69	8.459
600	350	518	9.5	61.89	7.128

Table 5: Sulfur specifications result from second sample reading: after 7 days stored at 25ºC room temperature.

Motor Speed (rpm)	Pump Rate (L/min)	Viscosity (cP)	pH	TSC (%)	D90 (μm)
800	750	402	8.3	60.88	7.988
800	550	502	8.8	62.13	6.662
800	350	548	9.0	62.41	6.617
700	750	330	9.3	60.28	8.807
700	550	514	9.4	61.22	8.010
700	350	512	8.3	60.98	6.303
600	750	260	9.5	58.92	9.854
600	550	312	9.4	60.30	8.482
600	350	426	8.2	59.40	7.221

Table 6: Sulfur specifications result from third trial run: after 22 hours stored at 70ºC oven temperature.

Motor Speed (rpm)	Pump Rate (L/min)	Viscosity (cP)	pH	TSC (%)	D90 (μm)
800	750	982	8.8	58.86	13.693
800	550	1082	8.8	60.56	12.052
800	350	1698	9.2	63.85	12.663
700	750	674	9.3	60.92	15.586
700	550	976	9.3	63.68	14.710
700	350	1090	8.8	60.44	10.057
600	750	578	9.3	59.65	21.075
600	550	784	9.2	62.90	18.391
600	350	1006	8.8	63.55	11.895

For each responses (viscosity, pH, TSC and particles size, D90), the average of the three trial runs were calculated and tabulated as in Table 7 through Table 10. The standard deviation and signal-to-noise (S/N) ratio were also calculated alongwith the average. In this study, the quality characteristic is smaller-the-better for means andlarger-the-better for S/N ratio were adopted.

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Table 7: Experimental results for sulfur dispersion viscosity

A Motor Speed	B Pump Rate	Viscosity I	Viscosity II	Viscosity III	Mean	Standard Deviation	S/N Ratio
800	750	472	402	982	618.667	316.596	5.81901
800	550	610	502	1082	731.333	308.450	7.49862
800	350	598	548	1698	948.000	650.000	3.27790
700	750	470	330	674	491.333	172.989	9.06713
700	550	646	514	976	712.000	237.966	9.51929
700	350	616	512	1090	739.333	308.106	7.60280
600	750	370	260	578	402.667	161.497	7.93562
600	550	432	312	784	509.333	245.319	6.34542
600	350	518	426	1006	650.000	311.718	6.38303

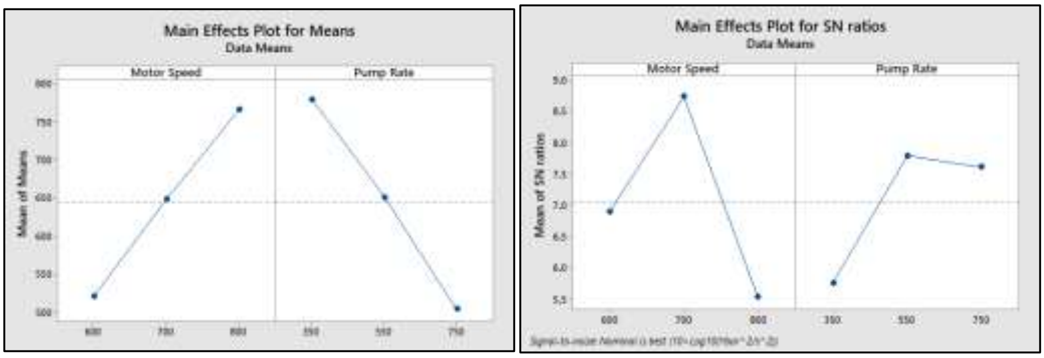

Figure 2: Effect of grinding mill parameters on viscosity for means and S/N ratio.

Table 8: Experimental results for sulfur dispersion pH

A Motor Speed	B Pump Rate	pH I	pH II	pH III	Mean	Standard Deviation	S/N Ratio
800	750	9.5	8.3	8.8	8.86667	0.602771	23.3522
800	550	9.5	8.8	8.8	9.03333	0.404145	26.9862
800	350	9.6	9.0	9.2	9.26667	0.305505	29.6381
700	750	9.7	9.3	9.3	9.43333	0.230940	32.2233
700	550	9.9	9.4	9.3	9.53333	0.321455	29.4425
700	350	9.5	8.3	8.8	8.86667	0.602771	23.3522
600	750	9.7	9.5	9.3	9.50000	0.200000	33.5339
600	550	9.7	9.4	9.2	9.43333	0.251661	31.4770
600	350	9.5	8.2	8.8	8.83333	0.650641	22.6557

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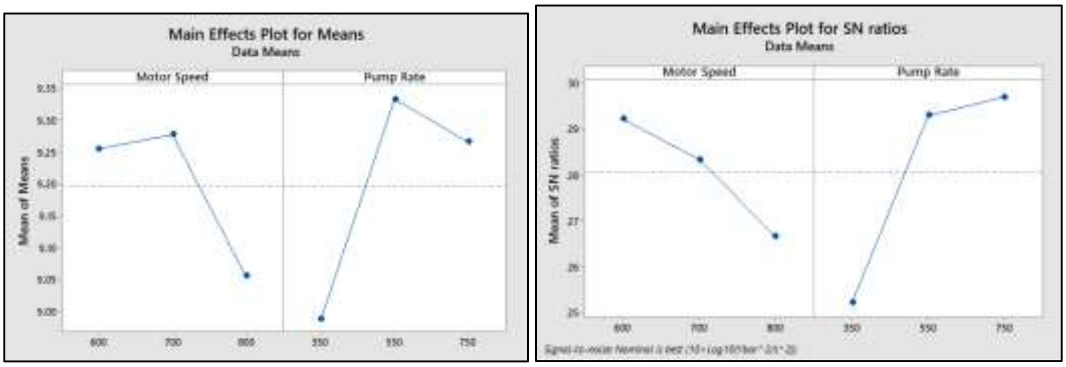

Figure 3: Effect of grinding mill parameters on pH for means and S/N ratio.

Table 9: Experimental results for sulfur dispersion TSC

A Motor Speed	B Pump Rate	TSC I	TSC II	TSC III	Mean	Standard Deviation	S/N Ratio
800	750	61.31	60.88	58.86	60.3500	1.30817	33.2803
800	550	62.99	62.13	60.56	61.8933	1.23217	34.0195
800	350	61.63	62.41	63.85	62.6300	1.12623	34.9031
700	750	62.05	60.28	60.92	61.0833	0.89623	36.6700
700	550	61.84	61.22	63.68	62.2467	1.27943	33.7420
700	350	62.31	60.98	60.44	61.2433	0.96241	36.0740
600	750	62.43	58.92	59.65	60.3333	1.85209	30.2579
600	550	62.69	60.30	62.90	61.9633	1.44431	32.6495
600	350	61.89	59.40	63.55	61.6133	2.08879	29.3956

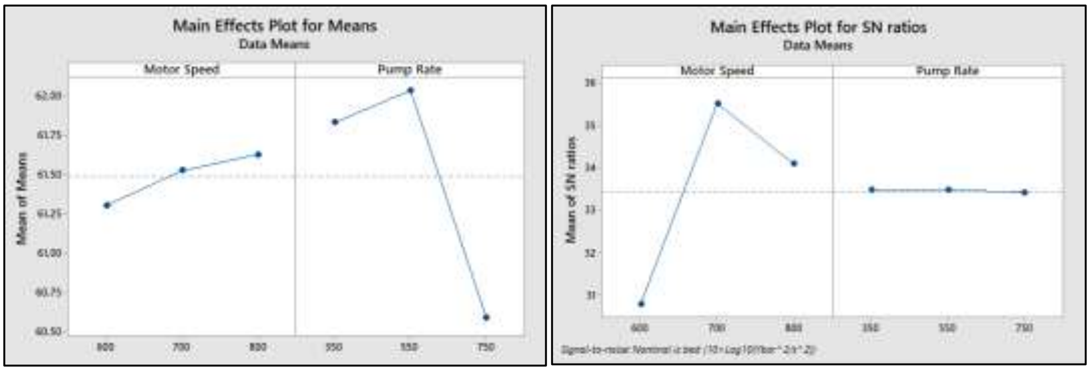

Figure 4: Effect of grinding mill parameters on TSC for means and S/N ratio.

Table 10: Experimental results for sulfur dispersion particles size, D90

A Motor Speed	B Pump Rate	D90 I	D90 II	D90 III	Mean	Standard Deviation	S/N Ratio
800	750	7.571	7.988	13.693	9.7507	3.42052	9.0988
800	550	6.840	6.662	12.052	8.5180	3.06183	8.8871
800	350	6.183	6.617	12.663	8.4877	3.62245	7.3957
700	750	8.558	8.807	15.586	10.9837	3.98768	8.8005
700	550	7.660	8.010	14.710	10.1267	3.97314	8.1267

700	350	6.902	6.303	10.057	7.7540	2.01682	11.6972
600	750	10.066	9.854	21.075	13.6650	6.41812	6.5640
600	550	8.459	8.482	18.391	11.7773	5.72761	6.2615
600	350	7.128	7.221	11.895	8.7480	2.72578	10.1284

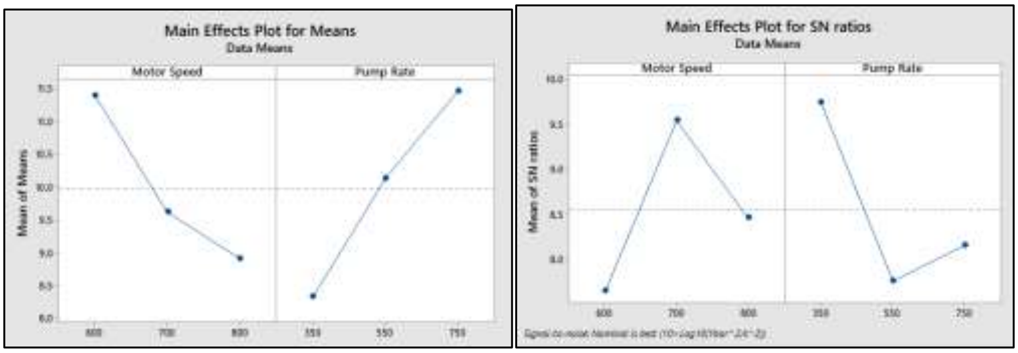

Figure 5: Effect of grinding mill parameters on particles size, D90 for means and S/N ratio.

Figure 2to Figure 5are the graphical information on the responses of both factors towards their respective means and S/N ratio. From the data and graphs, it can be seen that as mentioned on using smaller-the-better for means and larger-the-better for S/N ratio, all the responses are affected tremendously. However, seeing at the most predominant response – particle size – the optimal motor speed can be seen at higher motor speed of 700 rpm or 800 rpm. This can be proven by seeing on smaller means and higher S/N ratio respectively on the particle size effect. Theoretically, a frequent collision of milling beads with sulfur results in lower particle size and shall be more stable over the time. Concurrently, lower pump rate of sulfur dispersion towards the horizontal agitator bead mill machine shall give more time for the frequent beads collision with sulfur particles as proven in larger S/N ratio at 350 L/min rate. Intuitively, pH and TSC are not much affected by these factors due to;(i) less correlation proven between pH and TSC effects from mechanical milling, and (ii) the changes in pH reading and TSC are most probably due to temperature conditions set as well asmanual measurement and calculation of TSC that somehow prone to human errors, respectively.

For the sulfur dispersion as in industrial applications, higher motor speed of milling sulfur dispersion at lower pump rate such as between 700 to 800 rpm and 350L/min respectively in a relatively 60L milling capacitycan be utilised to obtain the desired product that able to meet their specifications.Apparently in this study, the Six Sigma approach is used to avoid some unnecessary abnormalities according to Taguchi's analysis in order to practice continuous improvement and enhancement.

Conclusion Remarks

The optimization analysis by using Six Sigma's Taguchi methodhas been successfully done in this study; with sulfur dispersion grinding mill motor speed of 800, 700 and 600 rpm, and pump rateof 750, 550 and 350 L/minas factors.The sulfur dispersion specifications or responses such as viscosity, pH, TSC and particles size, D90 were also tabulated and analysed. It can be drawn that the milling process parameters were optimized at relatively high milling motor speed (700 to 800 rpm) and at lowpump rate (350 L/min). By plotting the graph of main effects plot for means and S/N ratio, it can be seen that all the sulfur chemical specifications have been influenced during the process, but most preferably the particle size of the dispersion.

All in all, this method is a useful optimization tool in any industries that performed various parameters in their processes so that they can increase productivity and lessen their products' defects as well.

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