

Optimization of ABS Latex Coagulation using Taguchi Method and Mathematical Modeling for Percent Particles Size Distribution

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Abstract

In this paper, the optimization of particles size distribution (PSD) in the coagulation processes of Acrylonitrile-Butadiene-Styrene (ABS) latex was studied using H₂SO₄ as coagulant agent. The effects of parameters such as; temperature, agitation speed, coagulant agent concentration, the ratio of latex to coagulant agent and residence time on coagulation process were investigated and optimized using Taguchi experimental design. Based on Taguchi method, sixteen experiments were designed and performed to get optimum conditions of effective factors. The optimum conditions for the selected factors were obtained as: temperature (95°C); coagulant concentration (2% wt); residence time (10 min); agitation speed (180 rpm) and latex to coagulant volume ratio (1:1). According to the results, latex to coagulant volume ratio was the main factor having significant effects on the particles size distribution of the ABS. In the optimal conditions, coagulated particles with narrow in particles size distribution (220-280 μm) were prepared. An equation with 5 main parameters for predicting particles size distribution in the range of 220-280 μm with good compatibility and correlations between its results and experimental data was derived.

Keywords: Acrylonitrile-Butadiene-Styrene(ABS); Coagulation; Taguchi; Mathematical modeling.

Introduction

Coagulation is one of the important processes in the polymer production. Polymer latex made from emulsion polymerization often needs to be converted into dry powder by coagulation. Graft copolymer comprising an elastomeric base polymer such as poly butadiene and grafted chains of a thermoplastic, non-elastomeric polymer such as styrene-acrylonitrile are coagulated for isolation from aqueous emulsion. The ABS polymer which is based on three monomers; acrylonitrile (A), Butadiene (B) and Styrene (S); represent the industrially most important thermoplastic two-phase systems with an amorphous structure [1,2].

ABS latex coagulation process is a post processing stage in the manufacturing of ABS resins where the particles are recovered from colloidal latex prepared by emulsion polymerization. Since the surfaces of the colloidal particles in the latex are negatively charged by adding emulsifiers in the polymerization step, the latex is a stable colloidal solution by electrostatic repulsion between particles. The particles begin to coagulate into bigger flocs in the coagulator where the repulsive forces are weakened by addition of a coagulant such as sulfuric acid [3].

Basically, coagulation and flocculation can be done by double layer compression, charge neutralization, bridging and colloid entrapment [4]. Coagulation will occur if the kinetic energy of the particles is sufficiently high to overcome the potential energy barrier. Energy barrier is the sum of the Vander Walls attraction energy and the electrostatic repulsion energy [5]. A general rule in the coagulation process is that the Zeta potential of the particles which is a good approximation of surface potential is less than 14 mV [6]. Coagulation is the process of encounters between two or more particles by transport mechanisms, such as Brownian motion, field shear, etc., and coalescence of these particles due to short range of interfacial forces [7]. The coagulation process thus modifies the size distribution of the suspended particles in dispersive systems and controls the behavior of suspensions. The particle-particle interaction leading to any such coagulation is one of the major factors determining the design parameters in latex processing operation.

Several techniques such as chemical, evaporative, shear and freeze coagulation have been developed to destabilize polymeric emulsions and cause the desired coagulation [8,9]. Rapid and controlled coagulation of latex, induced by chemical coagulant, mechanical shear and heat, has been found to be the most effective process for the recovery of polymer from the latex. Chemical coagulation is the most common technique for coagulation, and is done by adding dilute salt or acid solution such as NaCl, MgSO₄, CaCl₂, Al₂(SO₄)₃ and H₂SO₄ to the colloidal solutions of polymers [10].

A major concern in the ABS latex processing is the excessive content of fine particles in the slurry coming out of the coagulator tank. The fine particles may be lost in the downstream recovering process or may lead to troubles in resin powders recovering. Thus, major efforts for process improvement have been directed to the reduction of fine particles in the coagulated slurry. The mean particles size in latex is in the range of 1-500 nm and after the coagulation process, the solid state have a mean particles size in the range of about 100-500 μm. Powder structure, particle size and particle size distribution (PSD) can be affected some parameters such as coagulant concentration, the ratio of latex to coagulant, agitation speed, residence time and temperature [2-4,10,11].

In this paper, optimization of coagulation process in the emulsion preparation of ABS latex using sulfuric acid as coagulant agent was investigated and experimental conditions to obtain a narrow particle diameter distribution with good morphology are studied based on

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Taguchi experimental design [12]. Because of industrial problems such as filtering, color stability and finishing process, the mean particles size in the range of 220-280 μm are desired and was selected in this work.

Experimental

Apparatus and reagents

The equipment's used in coagulation process are shown in Figure 1. The experiments were conducted in a 500 mL beaker that equipped with a mixer by a blade shaft and Thermostat (Lauda RE 104) for temperature controlling. ABS latex used in this experiment was prepared from Tabriz Petrochemical Company. The coagulant was aqueous solution of H_2SO_4 (98%, Merck, Germany). NaOH (98%, Fluka, Switzerland) was used to adjust pH of the solutions. Particles size distribution (PSD) and morphology of the ABS resin were studied by images resulted from Scanning Electron Microscopy (SEM, VEGA TESCAN, HV 20.0 KV). Ventilation oven (Memmert, UFB, 230V) was used for drying the products.

Recommended procedure

According to the set up in Figure 2, experiments were initiated by mixing the H_2SO_4 solution and latex in 75°C for 10 seconds at 600 rpm to form uniform dispersion of the coagulant and latex. Coagulation then continued with mild stirring by adjustment the pH of solution in the range of 3-4 by addition of NaOH (10% wt) [3]. The final slurry is neutralized and treated at 80-95°C to obtain the solidified particles. At the end, stirring was stopped and coagulated particles were collected by filtration through a 200 μm mesh size filter and dried in a ventilation oven at 30°C for about 24 hours. Images of the prepared ABS particles were obtained by SEM and measurement-Nano software was used for determination of particles size distribution.

Results and Discussion

Design of the experiment by Taguchi method

According to Taguchi experimental design, we can assume independent behavior of all effecting input factors on the process. On the other hand, the effect of an independent variable on the performance parameter does not depend on the different levels of other independent variables [13].

According to the previous works on ABS latex coagulation process [2-4,7] and also many preliminary experiments, the main operational factors and their levels were selected which are shown in Table 1. Residence time, temperature, agitation speed, concentration of coagulant and latex to coagulant volume ratio were selected as factors which have more effect in the coagulation process of ABS latex. Normally, in the case of five factors with four levels it is necessary to take 1024 (4^5) experiments in the optimization process. By Taguchi method, the standard orthogonal array, namely L_{16} reduces the number of experiments to 16. The designed L_{16} is an array of 16 experiments with the specified combination of the levels of factors. Factors are in columns and each row determines the levels of factors which must be used to run every individual experiment. The structure of Taguchi's orthogonal robust design and the results of measurements are shown in Table 2. The last column in this table shows calculated signal to noise ratio (S/N) from the measured results. The S/N ratios are different according to the type of characteristic and are classified into three groups [13].

Type N (nominal is the best):

$$\eta = S/N_N = 10 \log \frac{\left(\frac{1}{n}\right) \left((\sum yi)^2 / n - (\sum yi^2 - (\sum yi)^2 / n) / (n-1) \right)}{(\sum yi^2 - (\sum yi)^2 / n) N - 1} \quad (1)$$

Type S (the smallest is the best):

$$\eta = S/N_S = -10 \log \frac{1}{n} (\sum yi \wedge 2) \quad (2)$$

Type B (the biggest is the best):

$$\eta = S/N_B = -10 \log \left(\frac{1}{n} \sum \frac{1}{yi \wedge 2} \right) \quad (3)$$

Where y_i is the characteristic property (PSD% in this study) and n is the number of measurements in each experiment. All S/N ratios in Table 2 were calculated according to the equation 3, the biggest is the best, in order to maximize the PSD in the range of 220-280 μm .

The average S/N ratios for each level of the parameters are summarized and are shown in Table 3. The average of S/N was calculated by comparing the average responses (η) obtained at different levels (j) for each factor. For example, average response of B at level 1 is obtained from the results of experiments 1, 5, 9 and 13 (Table 2).

$$M_{B1} = \frac{\eta_1 + \eta_5 + \eta_9 + \eta_{13}}{4} = 26.74 \quad (4)$$

Similar calculations led to: $M_{B2} = 27.57$, $M_{B3} = 28.34$ and $M_{B4} = 30.53$. Figure 3 shows the effects of each factor on PSD based on the average S/N. From these results, the optimum levels for each factors can be obtained as: A_3 , B_4 , C_1 , D_2 and E_4 (i.e. A at level 3, B at level 4, C at level 1, D at level 2 and E at level 4).

The results for term $L_{\max} - L_{\min}$ are shown in the last column of Table 3 which resembles the highest value for factor E. According to this result the volume ratio of latex to coagulant is the most important factor in the coagulation process.

Analysis of variance

The purpose of Analysis of variance (ANOVA) is to investigate which factor significantly affect the particles size distribution. This is accomplished by separating the total variability of the S/N ratio, which is measured by the sum of the squared deviations (SS) from the total mean of the S/N ratio (S_m), into contributions by each factor and the error. Equations for ANOVA calculations are as follows [13,14].

Total degree of freedom

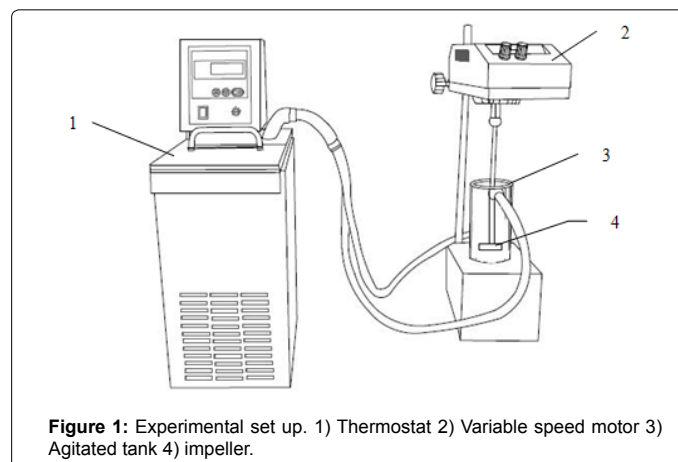


Figure 1: Experimental set up. 1) Thermostat 2) Variable speed motor 3) Agitated tank 4) impeller.

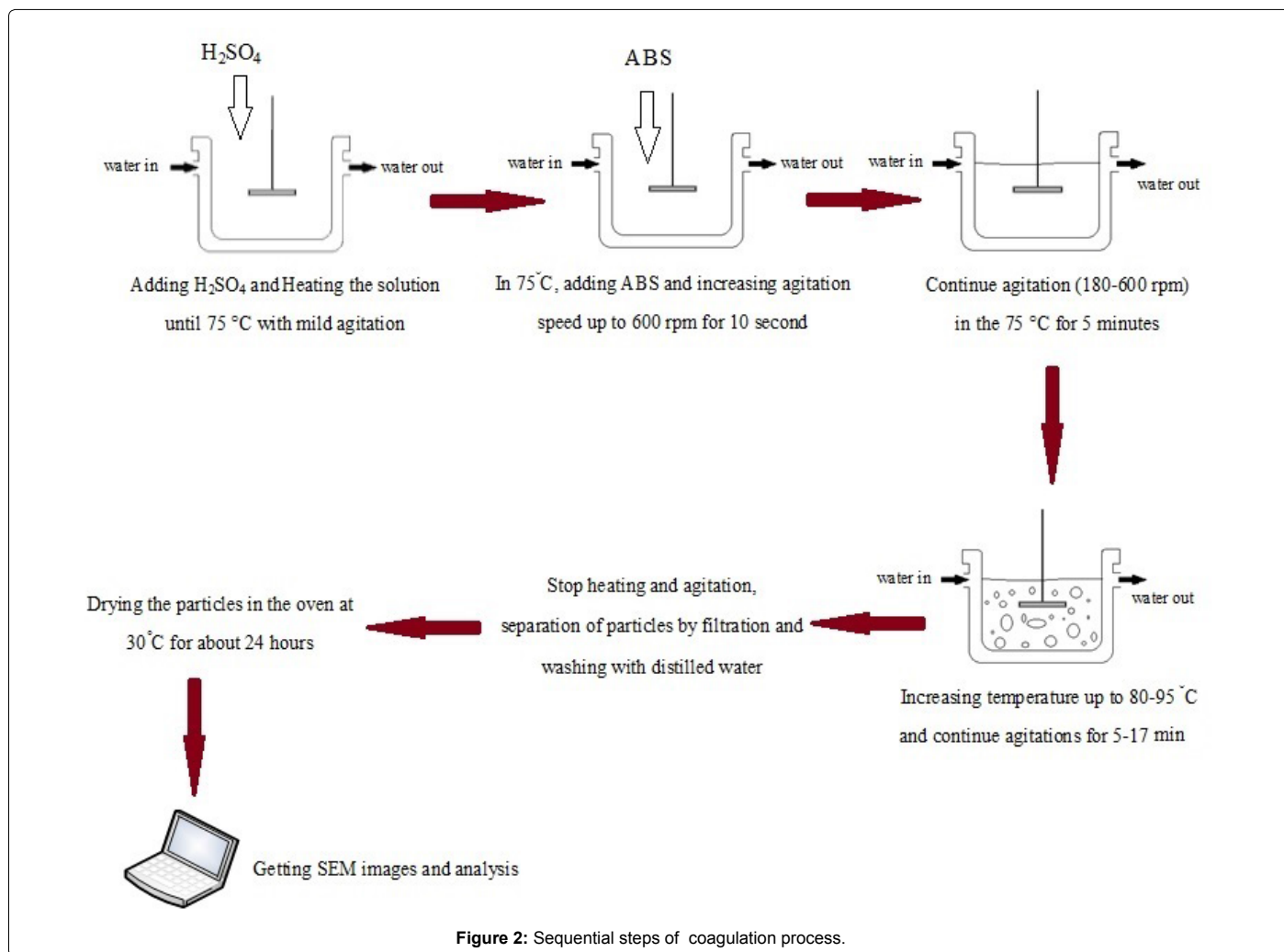


Figure 2: Sequential steps of coagulation process.

Parameter	Level1	Level 2	Level3	level4
A. Time (min)	5	7.5	10	17.5
B. Temperature (°C)	80	85	90	95
C. Agitation speed (rpm)	180	350	450	600
D. Coagulant concentration agent (wt %)	1	2	3	4
E. latex to coagulant Volume ratio	3:1	2:1	3:2	1:1

Table 1: Factors and their values corresponding to the levels in the experiments.

$$f_T = N - 1 = 16 - 1 = 15 \quad (5)$$

Where, N is the total number of result.

Degree of freedom for a factor

$$f_A = K_A - 1 = 4 - 1 = 3 \quad (6)$$

Where, K_A is the number of level for factor A and f_A is the degree of freedom of factor A.

Degree of freedom for error:

$$f_e = f_T - f_A - f_B - f_C - f_D - f_E \quad (7)$$

$$S_m = \text{Correction factor} = \frac{(\sum_{i=1}^{16} S/Ni)^2}{16} = (452.72)^2 \quad (8)$$

$$SS_i = \text{Sum of squares for factor I} = \frac{(4 \times m_{i1})^2 + (4 \times m_{i2})^2 + (4 \times m_{i3})^2 + (4 \times m_{i4})^2}{4} - S_m \quad (9)$$

Where, m_{i1} , m_{i2} , m_{i3} and m_{i4} are the average S/N ratio for level 1, 2, 3 and 4 of factor I, respectively.

$$S_T = \text{Total sum of squares} = \sum_{i=1}^{16} \left(\frac{S}{Ni}\right)^2 - S_m = 166.53 \quad (DF = 15) \quad (10)$$

$$MS_j = \text{Mean sum of squares for factor i} = \frac{SS_i}{DF \text{ of } i} \quad (11)$$

$$S_e = \text{Error sum of squares} = S_T - \sum SS_i \quad (12)$$

$$F_i = \text{Variance ratio for factor i} = \frac{MS_i}{MS \text{ of error}} \quad (13)$$

F_A, F_B, F_C, F_D, F_E and F_e is indeterminate since $V_e = 0$

$$P (\%) = \text{the percentage contribution for factor I} = \frac{SS_i - (DF \text{ of error})}{ST} \times 100 \quad (14)$$

The results for ANOVA calculations are shown in Table 4. According to the results, latex to coagulant volume ratio has the most effect on the coagulation processes. Temperature and coagulant concentration has the next largest effect on the coagulation process, respectively.

However, "error" could not be calculated directly (degree of freedom for error was obtained zero), therefore the "Pooling technique"

Parameter Exp. No.	A	B	C	D	E	Res ults , PSD% (220-280 μm)			
						Run1	Run 2	Average of	S/N
						results			
1	5	80	180	1	3:1	21.81%	18.46%	20.13%	25.99
2	5	85	350	2	2:1	22.53%	26.08%	24.30%	27.64
3	5	90	450	3	3:2	24.52%	30.76%	27.64%	28.66
4	5	95	600	4	1:1	40.47%	33.33%	36.90%	31.22
5	7.5	80	350	3	1:1	31.66%	25.80%	28.73%	29.03
6	7.5	85	180	4	3:2	24.24%	25.81%	25.02%	27.95
7	7.5	90	600	1	2:1	23.28%	23.33%	23.30%	27.35
8	7.5	95	450	2	3:1	29.72%	24.24%	26.98%	28.49
9	10	80	450	4	2:1	16.77%	16.29%	16.53%	24.36
10	10	85	600	3	3:1	17.00%	13.13%	15.06%	23.34
11	10	90	180	2	1:1	60.31%	51.51%	55.91%	34.87
12	10	95	350	1	3:2	42.31%	48.48%	45.39%	33.08
13	17.5	80	600	2	3:2	22.22%	26.00%	24.11%	27.56
14	17.5	85	450	1	1:1	36.84%	36.95%	36.89%	31.34
15	17.5	90	350	4	3:1	11.62%	16.21%	13.91%	22.51
16	17.5	95	180	3	2:1	31.82%	27.27%	29.54%	29.33

Table 2: Experimental layout using the L16 orthogonal array and experimental results for percentage particles size distribution and S/N ratio.

Factor	Level 1	Level2	Level 3	level 4	Lmax-Lmin
A. Time (min)	28.38	28.20	28.91	27.69	1.22
B. Temperature (°C)	26.74	27.57	28.34	30.53	3.79
C. Agitation speed (rpm)	29.54	28.07	28.21	27.36	2.17
D.Coagulant concentration (wt%)	29.44	29.64	27.59	26.51	3.13
E. Latex to coagulant volume ratio	25.08	27.17	29.31	31.61	6.53

Table 3: Average of the S/N ratios for each level of the factors.

is used to estimate the approximate error. By pooling, residence time is excluded as a factor that has minimum effect on the PSD. The results of pooled calculations are shown in Table 5.

Prediction and confirmation of the optimum conditions

The final step in the Taguchi method is predicting and verifying the improvement of the quality characteristic using the optimal levels of the factors. The predicted S/N ratios using the optimal levels of the factors can be calculated as follows [12,13]; predicted $S/N = S/N_m + \{(S/N_1 - S/N_m) + (S/N_2 - S/N_m) + (S/N_3 - S/N_m) + (S/N_4 - S/N_m) + (S/N_5 - S/N_m)\}$ Where S/N_m is the total mean S/N ratio and $S/N_1, S/N_2, S/N_3, S/N_4, S/N_5$ is the mean S/N ratio at the optimal level. For PSD in the desired range, the calculated value of S/N_m from Table 2 is 28.30. Also, S/N ratio for A3, B4, C1, D2 and E4 can be obtained from Table 3 and the values are 28.91, 30.53, 29.54, 29.64 and 31.61, respectively. Using these values, the above equation can be written as predicted $S/N = 28.30 + \{(28.91 - 28.30) + (30.53 - 28.30) + (29.54 - 28.30) + (29.64 - 28.30) + (31.61 - 28.30)\}$. Therefore, the predicted S/N ratio (37.095) for desired PSD can then be obtained and the corresponding estimated PSD can also be calculated by the following equation; $37.05 = -10 \log \frac{1}{y_i^2}$. Table 6 shows the comparison of the predicted PSD and the experimental results using the optimum conditions. The results show good agreement between the predicted and experimental PSD. The SEM images of the obtained particles under the optimum conditions are shown in Figure 4 in which the sizes of the particles are in the desired ranges (220-280 μm).

Effect of pH

In order to investigate the effect of pH, many experiments were done in the different levels of pH. Best results were obtained in the

pH levels between 3 and 4 in the coagulation process. In the high pH value, the latex could not coagulate completely because Zeta potential and electrostatic repulsion between particles were high [6]. In low pH value, rapid coagulation will take place and the coagulated particles take an irregular shape.

Effect of coagulant concentration

The concentration of coagulant agent has more effect on the coagulation process. In the high concentration of coagulant agent, pH is less than 2, thus particles exceed the energy barrier. In this case rapid coagulation occurs and consequently particles have poor morphology with wide diameter distribution and so higher amount of fine particles were produced. On the other hand, when the coagulant concentration is low, coagulated particles are small in size because electrostatic repulsion between particles is high. In the coagulant concentration slightly less than the critical coagulant concentration (CCC), the rate of coagulation is decreased and mixing rate can be used to control aggregated size and shape [15], but in these experiments it is found that high concentration of coagulant agent cause quick coagulation process and so there is not special effect from the mixing rate. According to the result which was shown in Figure 3A and Table 3, the optimum coagulant concentration was obtained as 2 wt%.

Effect of temperature

Temperature of the solution is the important parameter in the coagulation process. Coagulated particles with the required diameter could be obtained at a suitable temperature and it is usually 10°C below the glass transition temperature, Tg [3]. Glass transition temperature of ABS is about 102°C [8]. It was found that the average diameter of particles was increased with increasing the operating temperature. More

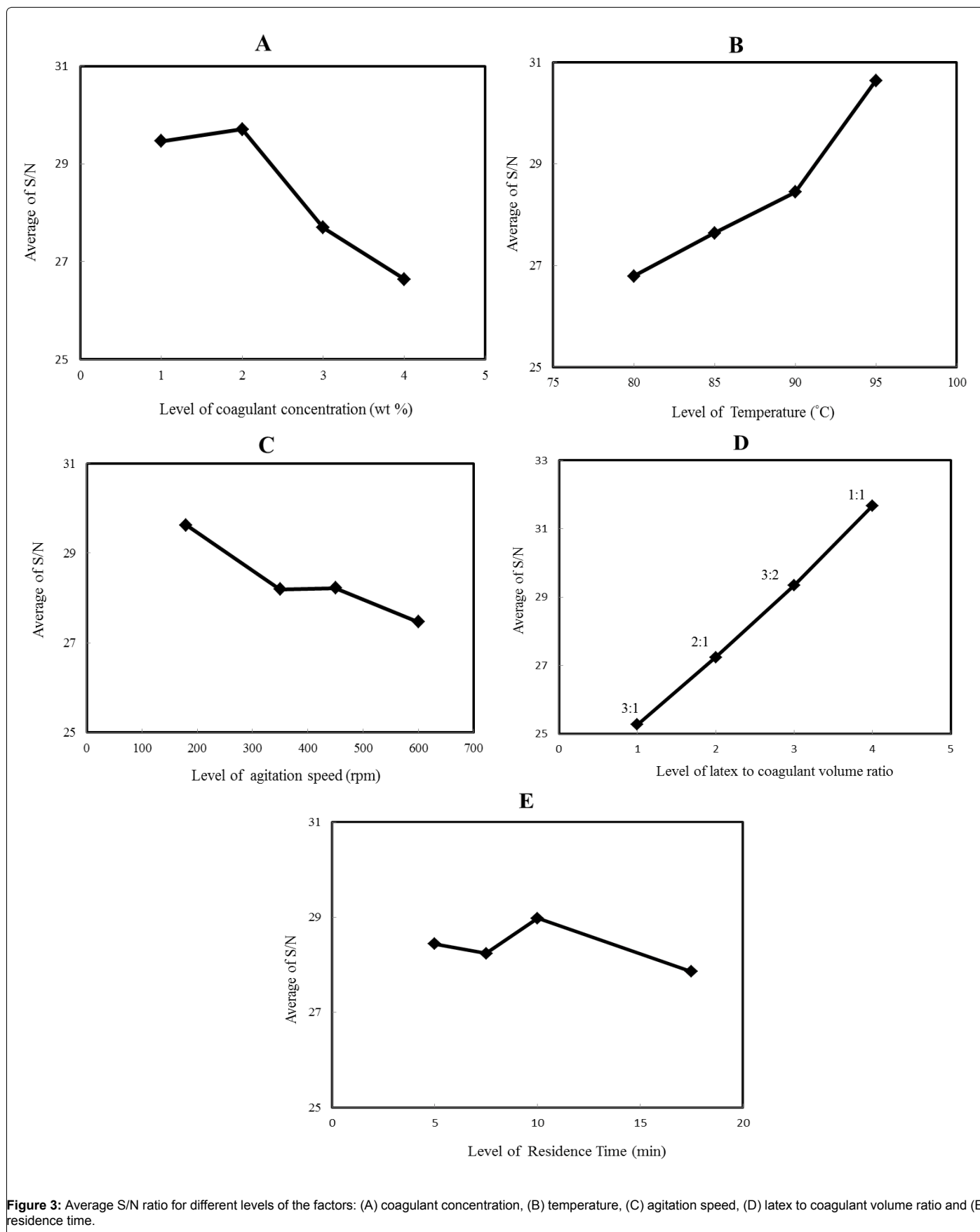


Figure 3: Average S/N ratio for different levels of the factors: (A) coagulant concentration, (B) temperature, (C) agitation speed, (D) latex to coagulant volume ratio and (E) residence time.

Factor	DF	SS	MS	F -Ratio	Pure s um S''	P%
A. Time(min)	3	3.07	1.02	----	3.70	1.84
B. Temperature(°C)	3	31.79	10.60	----	31.79	19.10
C. Agitation s peed (rpm)	3	9.83	3.28	----	9.83	5.91
D. Coagulant concentration (%wt)	3	27.16	9.05	----	27.16	16.32
E. Latex to coagulant volume ratio	3	94.55	31.52	----	94.55	56.82
Total	15	166.40				100%

Table 4: ANOVA res ults for S/N ratio of the PSD.

Factor	DF	SS	MS	F -Ratio	Pure s um S''	P%
A. Time(min)	(3)	(3.07)	1.02	Pooled	----	----
B. Temperature(°C)	3	31.79	10.60	10.36	28.72	17.26
C. Agitation s peed (rpm)	3	9.83	3.28	3.20	6.76	4.06
D. Coagulant concentration (%wt)	3	27.16	9.05	8.85	24.10	14.48
E. Volume ratio of latex to coagulant	3	94.55	31.52	30.80	91.48	54.97
Error	3	3.07	1.02			9.23
Total	15	166.40			100%	

Table 5: Pooled ANOVA res ults for S/N ratio of the PSD.

	Levels	PSD (%)	S/N ratio
Prediction	A3,B4,C1,D2,E4	71.25	37.05
Confirmation	A3,B4,C1,D2,E4	70.88	37.01

Table 6: Prediction and confirmation res ults for PSD.

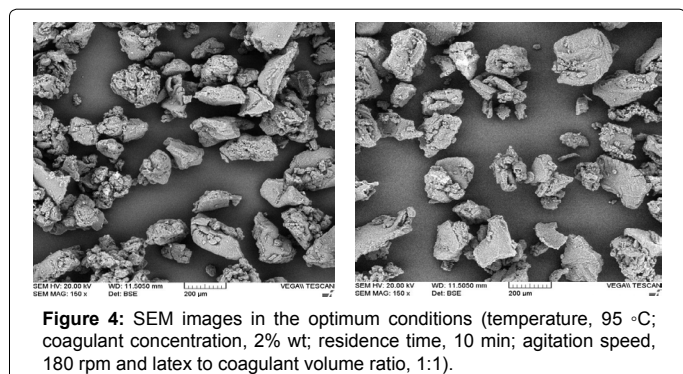


Figure 4: SEM images in the optimum conditions (temperature, 95 °C; coagulant concentration, 2% wt; residence time, 10 min; agitation speed, 180 rpm and latex to coagulant volume ratio, 1:1).

flocs appeared with increasing the temperature and thus the resulted particles were larger. When interfacial stress increases and Brownian motion speeds up with increasing temperature, latex particles are then easy to form with an irregular shape and loose structure [7]. From the result which was shown in Figure 3B and Table 3, the optimum temperature for coagulation of ABS was obtained as 95°C.

Effect of agitation speed

High agitation speed provides more favorable conditions for breaking up the particles than for coagulation. The size of particles is governed by the equilibrium of the attractive forces among the latex particles and the shear stress provided by the blades. The average diameter of the particles becomes smaller with increasing the agitation speed. According to results from Figure 3C and Table 3 the optimum agitation speed was obtained as 180 rpm

Effect of latex to coagulant volume ratio

The volume ratio of latex to coagulant agent has the most important role in the coagulation process. Reduction of this ratio leads to high PSD in the desired size. Increase of the slurry content in the latex leads to the formation of more particles. In this condition, polymer content increases with low energy dissipation but increase the content of fine and smaller coagulated particles with a wide distribution in size. From the results which are shown in Figure 3D and Table 3, the 1:1 ratio was obtained as the optimum latex to coagulant volume ratio.

Effect of residence time

Distribution of the particles diameter is affected by residence time because the attractive forces between the coagulated particles are weak and the particles are easily broken. So, the diameter of particles becomes larger with increasing the residence time. According to results from Figure 3E and also Table 3 the optimum residence time was obtained as 10 min.

Mathematical Model

Driving an equation according to the results of the optimized parameters could be useful in prediction of PSD%. From the results of 16 experiments and Data fit software equation 15 was derived as a result of mathematical modeling.

$$Y = \exp [a * X1 + b * X2 + c * X3 + d * X4 + e * X5 + f] \quad (15)$$

The results related to modeling calculations are shown in Table 7 and Figure 5. Variables and their values for derived equation are also shown in Table 8. This model could be used to predict percent particles size distribution in the desired size range (220-280 µm) as a function

	PSD%	Agitation speed (rpm) (X5)	Volume ratio (latex/coagulant) (X4)	Coagulant Conc. (%wt) (X3)	Temperature (°C) (X2)	Time (min) (X1)
Run 1	20.13	180	3:1	1	80	5
Run2	24.30	350	2:1	2	85	5
Run3	27.64	450	3:2	3	90	5
Run4	36.90	600	1:1	4	95	5
Run5	28.73	350	1:1	3	80	7.5
Run6	25.02	180	3:2	4	85	7.5
Run7	23.30	600	2:1	1	90	7.5
Run8	26.98	450	3:1	2	95	7.5
Run9	16.53	450	2:1	4	80	10
Run10	15.06	600	3:1	3	85	10
Run11	55.91	180	1:1	2	90	10
Run12	45.39	350	3:2	1	95	10
Run13	24.11	600	3:2	2	80	17.5
Run14	36.89	450	1:1	1	85	17.5
Run15	13.91	350	3:1	4	90	17.5
Run16	29.54	180	2:1	3	95	17.5

Table 7: Data fit results for percentage particles size distribution in different levels of the factors.

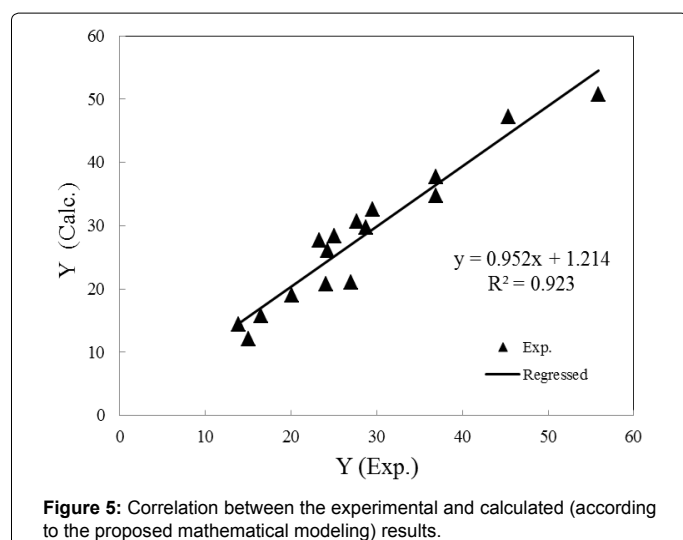


Figure 5: Correlation between the experimental and calculated (according to the proposed mathematical modeling) results.

Variable	Value
a	-5.8338577032605E-03
b	3.02411611742547E-02
c	-0.114074699106494E-04
d	-0.407078317729991
e	-7.51809709863315E-04
f	2.02365206866783

Table 8: Values for the variables in the proposed mathematical equation.

of the factors according to Table 1. Prediction results obtained by this equation have 92% matches by the experimental results.

Conclusion

Coagulation process of ABS was performed using H_2SO_4 as chemical

coagulant agent and Taguchi method was used for optimizing the effective factors to obtain desired characteristics for particles sizes. The effects of various factors on the particles size distribution were studied and optimized conditions were obtained. According to the result, latex to coagulant volume ratio was the main factor having significant effects on the particles size distribution of the ABS. In the optimal conditions, coagulated particles with narrow in particle size distribution (220-280 μm) were prepared. An equation with 5 main parameters for predicting particles size distribution in the range of 220-280 μm with good compatibility and correlations between its results and experimental data was derived.

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Abbreviations

- Particles Size Distribution (PSD)
- Acrylonitrile-Butadiene-Styrene (ABS)
- Scanning Electron Microscopy (SEM)
- Critical Coagulant Concentration (CCC)
- Glass Transition temperature (Tg)

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