Application of Factorial Experimental Design to Study the Influence of Polymerization Conditions on the Yield of Polyaniline Powder

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ABSTRACT: Influences of chemical oxidation polymerization conditions on the yields of polyaniline powder were investigated. These chemical oxidation polymerization conditions included polymerization time, concentration of HNO_3, mol ratio of ammonium persulfate/aniline (APS/AN), and polymerization temperature. If polymerization time, concentration of HNO_3, mol ratio of APS/AN, and polymerization temperature were 60 min, 1.0 *M*, 1.0, and 0°C, respectively, then the yield of emeraldine base form polyaniline powder was around 78.1%. The yield of polyaniline powder increased significantly with the polymerization time, concentration of HNO_3, and the mol ratio of APS/AN. A 2^3 factorial experimental design was applied to study the main, two-factor interaction, and three-factor interaction effects of polymerization time, concentration of HNO_3, and mol ratio of APS/AN on the yield of polyaniline powder. According to the definition, the sequence of the main effects on the yield of polyaniline powder, in ascending order, is concentration of HNO_3 < mol ratio of APS/AN < polymerization time. The sequence of the two-factor interaction effects on the yield of polyaniline, in ascending order, is concentration of HNO_3 vs. mol ratio of APS/AN < polymerization time vs. concentration of HNO_3 < polymerization time vs. mol ratio of APS/AN. Meanwhile, the prediction equation by definition is: $\hat{Y} = 0.287+0.145X_1+0.091X_2+0.121X_3+0.023X_1X_2+0.111X_1X_3+0.002X_2X_3+0.003X_1X_2X_3$. © 2002 Wiley Periodicals, Inc. J Appl Polym Sci 85: 1571–1580, 2002

Key words: factorial experimental design; polyaniline; yield; emeraldine base; polymerization condition

INTRODUCTION

Experimental designs and their statistical analyses have been well developed and applied widely in many research areas, such as basic science, engineering, sociology, etc. The main advantage of the experimental design is that it can cover a larger area of engineers' experimental interest and obtain unambiguous results at a minimum cost.[1,2] Because this technique is powerful and easy to handle, the factorial experimental design is one of the most commonly used methods to realize the effects of some independent variables that significantly affect the final experimental results.

In 1993, Bambrick et al.[3] studied the fusion characteristics of poly(vinyl chloride) (PVC) compounds. The dependent variables are fusion time, fusion temperature, and fusion torque. They used a Rheocord System 40 torque rheometer equipped with a Rheomix 600 bowl and roller mixing blades. Moreover, they applied a central compos-

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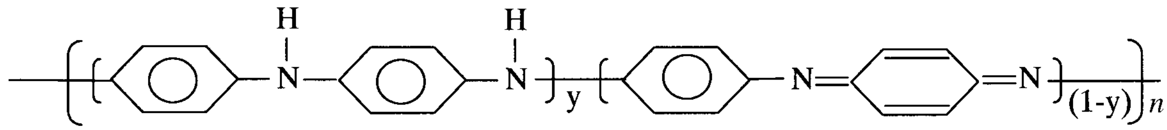
ite design (CCD) of the experiment to find the optimal formulation of additives for PVC compounds by changing the following six independent formulation variables: amounts of impact modifier, paraffin wax, calcium stearate, ester wax, and processing aid. In a previous work,[4] we applied a 2^3 factorial experimental design to study the main, two-factor interaction, and three-factor interaction effects of three independent blending conditions (starting temperature, rotor speed, and totalized torque), on the heat of fusion of PVC/CPE/OPE blends.

Currently, polyaniline has been an important member in the family of intrinsically conducting polymers (ICP). Because polyaniline has excellent environmental stability and unique electrochemical property, many applications of polyaniline have been studied and developed, such as secondary batteries,[5,6] biosensors,[7,8] corrosion protections,[9,10] antistatic packaging materials,[11] etc. MacDiarmid et al.[12] have illustrated that the chemical structure of polyaniline could be schematically represented by the following formula:



where the value of $(1-y)$ represents the oxidation state of polyaniline. The value of y can be varied from $y = 1$ (leucoemeraldine base: LEB) to $y = 0$ (pernigraniline base: PNB). If $y = 0.5$, the polyaniline is referred to as emeraldine base (EB) form polyaniline. This EB form polyaniline cannot be dissolved in common organic solvents. However, it can be dissolved in 1-methyl-2- pyrrolidinone (NMP). Then, free-standing EB form polyaniline films can be cast from the NMP solution.[13,14] Moreover, EB form polyaniline can be doped in a protonic acid such as HCl or H_2SO_4 and transferred to emeraldine salt (ES) from polyaniline with a moderately high conductivity up to 10–100 S/cm.

Generally speaking, chemical oxidization and electrochemical synthesis are two major routes for preparing polyaniline powder.[15,16] Chemical oxidation polymerization process is particularly important because this synthesis is the most feasible method to produce polyaniline powder on a large scale. Pron et al.[17] studied the relationship between the electrical conductivity and the yield of polyaniline powder, polymerized with four different oxidizing agents and at different aniline/oxidant ratios. They concluded that the redox potential of the oxidants is not a dominant factor in the chemical polymerization of aniline. Their reports showed most oxidizing agents with similar results. Armes et al.[18] concluded that the electrical conductivity, yield, elemental composition, and degree of oxidation of the resulting polyaniline powder were basically independent of the oxidant/monomer initial mol ratio when its value was below 1.15. They also reported that overoxidation of polyaniline powder occurs at higher oxidant/monomer initial mol ratios. Asturias et al.[19] illustrated the influence of the polymerization atmosphere (air or argon) on the degree of oxidation of chemically polymerized polyaniline powder by using ammonium persulfate (APS) as an oxidizing agent. Cao et al.[16] investigated the chemical polymerization of aniline in aqueous solutions that was studied as a function of a wide variety of synthesis parameters, such as pH, oxidizing agents, protonic acids, relative concentration of reactants, polymerization temperature and time, etc. They found that the reaction yield was not strongly sensitine to most synthesis variables, while the viscosity, molecular weight, and the electrical conductivity of the as-polymerized and/or posttreated polyaniline salt were found to be significantly affected. Recently, Ruckenstein et al.[20,21] prepared soluble polyaniline codoped with dodecyl benzene sulfonic acid (or camphor sulfonic acid) and hydrochloric acid by chemical oxidation in aqueous solution. They found that the conductivity and yield of the polymer were strongly dependent on the polymerization conditions, such as oxidant amount, polymerization temperature, concentration of HCl aqueous solution, etc.

In those works mentioned above, the acid media for polymerization were commonly HCl, H_2SO_4, $HClO_4$, etc. To our knowledge, very few systematic studies based on the polymerization medium of HNO_3 aqueous solution have been reported. Therefore, in this article, we systemically illustrated the influence of polymerization time, concentration of HNO_3, mol ratio of APS/AN, and polymerization temperature on the yield of polyaniline powder. We found that the polymerization time, concentration of HNO_3, and mol ratio of APS/AN were three important factors affecting the yield of polyaniline powder. Therefore, we applied a 2^3 factorial experimental design [three independent variables with high (+), and low (−) levels] to study the main, two-factor interaction, and three-factor interaction effects, of these three independent polymerization variables on the yield of polyaniline powder. A prediction equation is also illustrated here.

EXPERIMENTAL

Materials

Syntheses-grade aniline and ammonium persulfate [$(NH_4)_2S_2O_8$] were purchased from Merck. Nitric acid (70 wt %) was purchased from Union Chemical Works Ltd. Aniline was purified by distillation under reduced pressure prior to use. The other reagents were used as received.

Polymerization Experiment

In this article, polyaniline powder was prepared by chemical oxidation with ammonium persulfate based on the procedure described by Chen et al.[14] and Cao et al.[16] Two solutions were prepared previously. Solution A was 200 mL of 1 *M* HNO_3 aqueous solution containing 8.22 g aniline (0.44 *M*). Solution B was 200 mL of 1 *M* HNO_3 aqueous solution containing 20 g ammonium persulfate (0.44 *M*). The molar ratio of aniline to ammonium persulfate was 1:1. A 1000-mL four-necked flat bottom reactor was used to prepare polyaniline powder. A stirrer was put in the reactor to ensure proper mixing. Then, the reactor was kept under vigorous stirring. After that, solution A was poured into this reactor that was placed into an ice bath containing salt and equipped with a thermometer. After the temperature of solution A was cooled to 0°C, solution B was then added drop by drop into solution A over a period of 3 min. Because the oxidation of aniline is highly exothermic, the addition rate of solution B should be properly controlled to prevent any sharply temperature increasing due to the reaction. After 60 min, the precipitated dark green ES polyaniline was recovered from the reaction mixture. Then, this material was filtered and washed by using 400 mL distilled water until the filtrate was colorless. Furthermore, the precipitate was washed again with methanol until the methanol filtrate was colorless to remove oligomers and other byproducts. Then, the prepared ES form polyaniline was converted to EB form polyaniline by stirring with 400 mL 1 *M* NH_4OH solution at room temperature for another 24 h. At the end of stirring, the material was filtered and dried under dynamic vacuum at 60°C for 48 h. Finally, 6.42 g of the dark blue EB form polyaniline powder was obtained (78.1% yield).

For the purpose of realizing the influences of polymerization time, concentration of HNO_3, and mol ratio of APS/AN on the polymerization of aniline monomers, four groups of polymerization experiments were designed and described as below. I. Concentration of HNO_3, mol ratio of APS/AN, and polymerization temperature were set at 1 *M*, 1.0 and 0°C, respectively. Five different polymerization times, 15, 30, 60, 90, and 120 min, were conducted. II. Polymerization time, mol ratio of APS/AN, and polymerization temperature were set at 60 min, 1.0 and 0°C, respectively. Six various Concentrations of HNO_3, 0, 0.1, 0.5, 1.0, 2.0, and 4.0 *M*, were used. III. Polymerization time, Concentration of HNO_3, and polymerization temperature were set at 60 min, 1.0 *M* and 0°C, respectively. Six different mol ratios of APS/AN, 0, 0.25, 0.5, 1.0, 1.5, and 2.0, were used. IV. Polymerization time, concentration of HNO_3, and mol ratios of APS/AN were set at 60 min, 1.0 *M* and 1.0, respectively. Five different polymerization temperatures, 0, 10, 20, 30, and 40°C were used.

After the specified polymerization experiment was finished, the polyaniline powder dedoped in 1.0 *M* NH_4OH aqueous solution, was collected, dried, and weighed. The yield of polyaniline powder was defined as below:

$$\text{Yield of polyaniline powder} = \frac{B}{A} \times 100\%$$

where A represents 8.22 g aniline monomer and B represents the weight of polyaniline powder dedoped in 1.0 *M* NH_4OH aqueous solution.

Experimental Design

The influence of polymerization temperature was not significant; therefore, polymerization time (X_1), concentration of HNO_3 (X_2), and mol ratio of APS/AN (X_3) were chosen as the independent variables of the factorial experimental design. Two levels, high (+) and low (−), were also defined for each independent variable. Thus, a 2^3 factorial experimental design will have eight

Table I Influence of Polymerization Time on the Yield of Polyaniline Powder

Polymerization Time (Minutes)	Yield of Polyaniline (%)
0	0.0
15	22.0
30	36.6
60	78.1
90	79.2
120	78.4

Concentration of HNO_3 = 1.0 M; mol ratio of APS/AN = 1.0; polymerization temperature = 0°C.

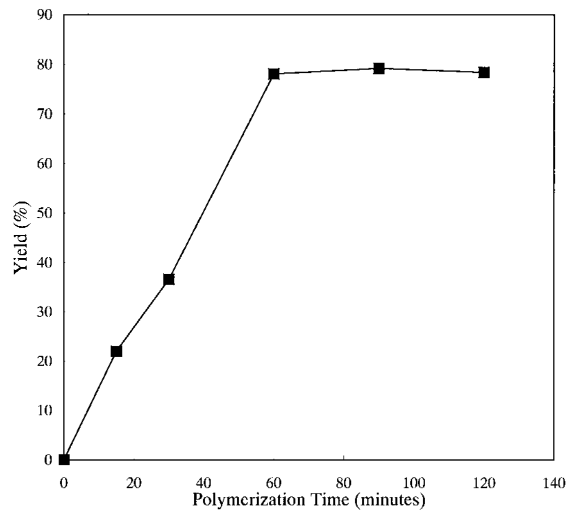


Figure 1 Influence of polymerization time on the yield of polyaniline powder (concentration of HNO_3 = 1.0 *M*, mol ratio of APS/AN = 1.0, and polymerization temperature = 0°C).

runs, the first in standard order being (−−−), and the last in standard order being (+++). For polymerization time, 60 and 15 min were chosen as high and low levels, respectively. For concentration of HNO_3, 1.0 and 0.25 *M* were chosen as high and low levels, respectively. For the mol ratio of APS/AN, 1.0 and 0.25 were chosen as high and low levels, respectively. The yield of polyaniline powder was studied as the dependent variable here.

RESULTS AND DISCUSSIONS

The influence of polymerization time on the yield of polyaniline powder was shown in Table I, and Figure 1. The concentration of HNO_3, mol ratio of APS/AN, and polymerization temperature were set at 1.0 *M*, 1.0, and 0°C, respectively. Generally speaking, the yield of polyaniline powder increased with polymerization time and eventually approaches steady yield when the polymerization time was longer than 60 min. In Figure 1, it indicated that the yield of polyaniline powder was almost proportional to polymerization time. If the polymerization time was extended to 60 min or longer, the yield of polyaniline powder was almost constant. It implied that the polymerization of aniline monomers was almost done in the first 60 min under the polymerization conditions as mentioned above.

Table II and Figure 2 indicated the relationship between the yield of polyaniline powder and the concentration of HNO_3. The polymerization time, mol ratio of APS/AN, and polymerization temperature were set at 60 min, 1.0, and 0°C, respectively. In Figure 2, when the concentration of HNO_3 was increased from 0.0 to 1.0 *M*, the yield of polyaniline powder increased from 42.0 to 78.1%, while with further increased to 4.0 *M*, the yield of polyaniline powder decreased to 59.2%. This indicates that high acidity accelerates hydrolysis of the polyemeraldine chains.[16] From this result we concluded that for chemical oxidative polymerization under APS oxidation, a HNO_3 concentration of 0.5–2.0 *M* resulted in higher yield of polyaniline powder. Cao et al.[16] studied the dependence of the molecular weight of polyaniline on the acidity of the reaction medium clearly and indicated that that two competing process, polymerization and degradation due to hydrolysis, controlled the polyaniline polymerization. They also found that not only the concentration but also the nature of the protonic acid, af-

Table II Influence of Concentration of HNO_3 on the Yield of Polyaniline Powder

Concentration of HNO_3 (*M*)	Yield of Polyaniline (%)
0	42.0
0.1	54.6
0.5	76.4
1.0	78.1
2.0	75.2
4.0	59.2

Polymerization time = 60 mins; mol ratio of APS/AN = 1.0; polymerization temperature = 0°C.

Table III Influence of mol Ratio of APS/AN on the Yield of Polyaniline Powder

Mol Ratio of APS/AN	Yield of Polyaniline (%)
0.0	0
0.25	31.0
0.5	52.6
1.0	78.1
1.5	79.2
2.0	51.8

Polymerization time = 60 min; concentration of HNO_3 = 1.0 M; polymerization temperature = 0°C.

Table IV Influence of Polymerization Temperature on the Yield of Polyaniline Powder

Polymerization Temperature	Yield of Polyaniline (%)
0.0	78.1
10.0	77.8
23.0	76.7
30.0	75.2
40.0	74.1

Polymerization time = 60 mins; concentration of HNO_3 = 1.0 M; mol ratio of APS/AN = 1.0.

fected the quality of the polymerization products. Moreover, MacDiarmid et al.[15] illustrated that the strong effect of the acidity of the polymerization medium on the electrical conductivity of synthesized polymer.

Table III and Figure 3 showed the influence of mol ratio of APS/AN on the yield of polyaniline powder. The polymerization time, concentration of HNO_3, and polymerization temperature were set at 60 min, 1.0 *M*, and 0°C, respectively. In Figure 3, when the mol ratio of APS/AN was increased from 0.0 to 1.0, the yield of mol ratio of APS/AN increased from 0.0 to 78.1%. When the mol ratio of APS/AN was increased to 1.5, the yield of polyaniline powder was slightly increased to 79.2%. However, if the mol ratio of APS/AN was increased to 2.0, the yield of polyaniline powder decreased to 51.8%. This indicates that at higher mol ratio of APS/AN, side reactions are expected to happen and the yield of polyaniline powder are expected to decrease with increasing amount of oxidant (APS).[20] From this result we concluded that for chemical oxidative polymerization under 1.0 *M* HNO_3 as polymerization medium, the mol ratio of APS/AN of 1.0–1.5 resulted in higher yield of polyaniline powder.

Table IV and Figure 4 showed the influence of polymerization temperature on the yield of polyaniline powder. The polymerization time, concentration of HNO_3, and mol ratio of APS/AN were set at 60 min, 1.0 *M*, and 1.0, respectively. In Figure 4, it indicated that the yield of polyaniline powder slightly decreased with increasing polymerization temperature. This happened because

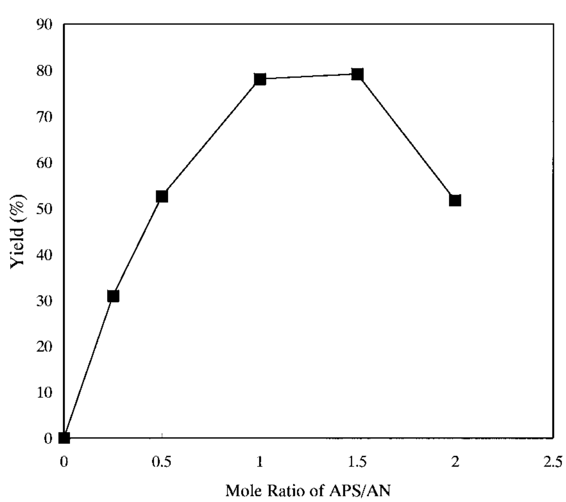


Figure 3 Influence of mol ratio of APS/AN on the yield of polyaniline powder (polymerization time = 60 min, concentration of HNO_3 = 1.0 *M*, and polymerization temperature = 0°C).

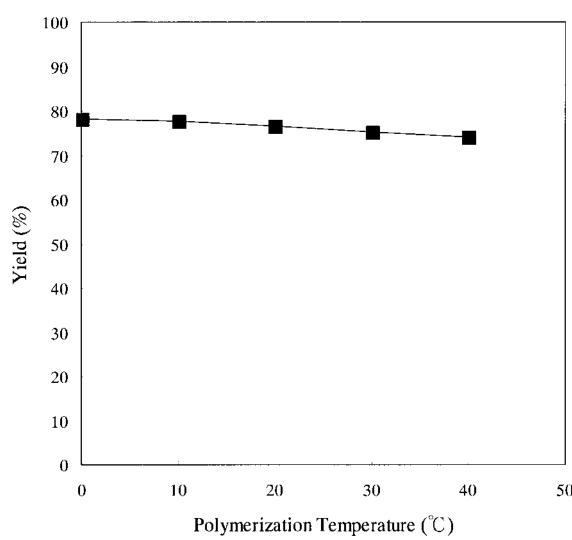


Figure 4 Influence of polymerization temperature on the yield of polyaniline powder (polymerization time = 60 min, concentration of HNO_3 = 1.0 *M*, and mol ratio of APS/AN = 1.0).

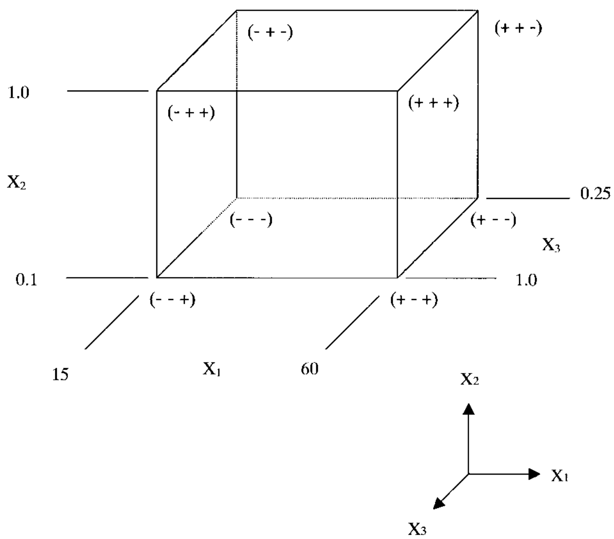


Figure 5 The diagrammatic representation of a 2^3 factorial experimental design standard ordering [Here X_1: polymerization time (minutes), X_2: concentration of HNO_3 aqueous solution (M), X_3: mol ratio of APS/AN].

the overoxidative and the hydrolysis side reactions were stimulated by higher temperature.[21]

Figure 5 shows the standard figure of a 2^3 factorial experimental design. Figure 6 shows the observed yields (the yield of polyaniline powder) and the standard ordering of polymerization experiments for aniline monomer. Figures 7, 8, and

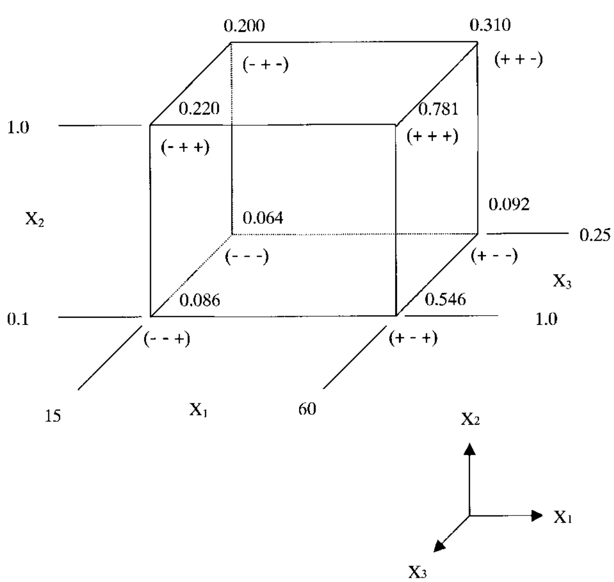


Figure 6 The diagrammatic representation of observed yields (the yield of polyaniline powder) and standard ordering of polymerization experiments for aniline monomer.

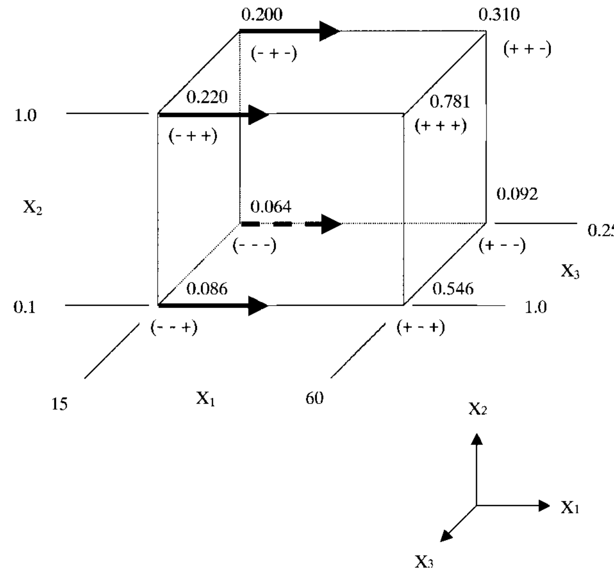


Figure 7 Determination of main effect of polymerization time (X1) on yield of polyaniline powder.

9 represent the determinations of the main effects of polymerization time, concentration of HNO_3, and mol ratio of APS/AN, respectively. According to the definition, the main effect of the controlled independent variable is the average of the difference between the values at the high level (+) and the values at the low level (−). Tables V–VII illustrate the results of the main effects of polymerization time, concentration of HNO_3, and mol

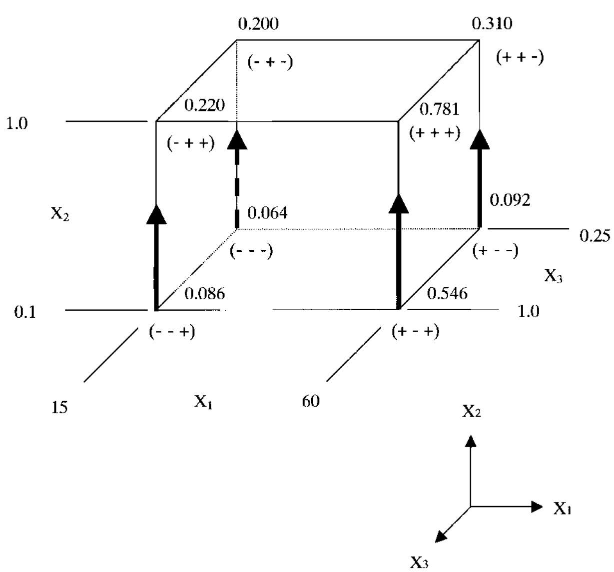


Figure 8 Determination of main effect of concentration of HNO_3 aqueous solution (X2) on yield of polyaniline powder.

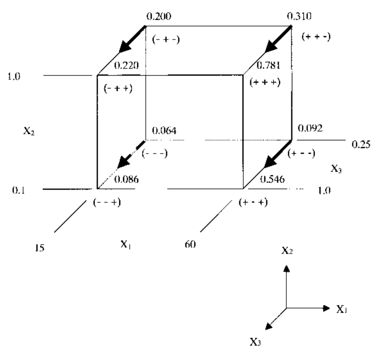


Figure 9 Determination of main effect of mol ratio of APS/AN (X_3) on the yield of polyaniline powder.

ratio of APS/AN, respectively. Comparing the results of these three tables, the sequence of the main effects on the yield of polyaniline powder, in ascending order, is the concentration of HNO_3 $<$ mol ratio of APS/AN $<$ the polymerization time.

Figures 10, 11, and 12 illustrate the determinations of polymerization time vs. concentration of HNO_3, polymerization time vs. mol ratio of APS/AN, and concentration of HNO_3 vs. mol ratio of APS/AN interaction effects, respectively. According to the definition, the two-factor interaction effect of polymerization time vs. concentration of HNO_3 (X_1 vs. X_2) is equal to half the difference [(0.336 − 0.244)/2 = 0.046] between the average polymerization time effect with concentration of 1.0 *M* HNO_3, [(0.561 + 0.110)/2 = 0.336], and the average polymerization time effect with a concentration of 0.1 *M* HNO_3, [(0.460 + 0.028)/2 = 0.244]. Polymerization time vs. mol ratio of APS/AN interaction effect (X_1 vs. X_3) is equal to half the difference [(0.511 − 0.069)/2 = 0.221] between the average polymerization time effect with a mol ratio of APS/AN = 1.0, [(0.561 + 0.460)/2 = 0.511], and the average polymerization time effect with a mol ratio of APS/AN = 0.25, [(0.110 + 0.028)/2 = 0.069]. Similarly, the concentration of HNO_3 vs. mol ratio of the APS/AN interaction effect (X_2 vs. X_3) is equal to half the difference [(0.185 − 0.177)/2 = 0.004] between the average concentration of HNO_3 effect with a mol ratio of APS/AN = 1.0, [((0.235 + 0.134)/2 = 0.185), and the average concentration of the HNO_3 effect with a mol ratio of APS/AN = 0.25, [(0.218 + 0.136)/2 = 0.177]. Consider the individual comparisons of the effect of polymerization time (X_1). There are two available measurements from the experiment to estimate the three-factor interaction effect, polymerization time vs. concentration of HNO_3 vs. mol ratio of APS/AN (X_1 vs. X_2 vs. X_3), one for each mol ratio of APS/AN, mol ratio of APS/AN = 1.0: [0.561 − 0.460)/2 = 0.15, mol ratio of APS/AN = 0.25: [0.110 − 0.028)/2 = 0.041. The difference between these two estimates is a measure of consistency for each concentration of HNO_3, concentration of 1.0 *M* HNO_3: (0.561 − 0.110)/2 = 0.226, and concentration of 0.1 *M* HNO_3: {0.460 − 0.028)/2 = 0.216. Half this difference, (0.051 − 0.041)/2 = 0.005 or (0.226 − 0.216)/2 = 0.005, is defined as the three-factor interaction effect of polymerization time vs. concentration of HNO_3 vs. mol ratio of APS/AN (X_1 vs. X_2 vs. X_3).

Table VI The Main Effect of Concentration of HNO_3 (X_2) on the Yield of Polyaniline Powder Conditions Where Comparisons Are Made

Effect of X_2 Individual Comparisons	X_1	X_3
(0.781–0.546) = 0.235	120	1.0
(0.220–0.086) = 0.134	15	1.0
(0.310–0.092) = 0.218	120	0.0
(0.200–0.064) = 0.136	15	0.0

Average (main effect of concentration of HNO_3): (0.235 + 0.134 + 0.218 + 0.136)/4 = 0.181.

Table V The Main Effect of Polymerization Time (X_1) on the Yield of Polyaniline Powder Conditions Where Comparisons Are Made

Effect of X_1 Individual Comparisons	X_2	X_3
(0.781–0.220) = 0.561	1.0	1.0
(0.546–0.086) = 0.460	0.1	1.0
(0.310–0.200) = 0.110	1.0	0.0
(0.092–0.064) = 0.028	0.1	0.0

Average (main effect of polymerization time): (0.561 + 0.46 + 0.11 + 0.028)/4 = 0.290.

Table VII The Main Effect of Mol Ratio of APS/AN (X_3) on the Yield of Polyaniline Powder Conditions Where Comparisons Are Made

Effect of X_3 Individual Comparisons	X_1	X_2
(0.781–0.310) = 0.471	120	1.0
(0.546–0.092) = 0.454	120	0.1
(0.220–0.200) = 0.020	15	1.0
(0.086–0.064) = 0.022	15	0.1

Average (main effect of mol ratio of APS/AN): (0.471 + 0.454 + 0.020 + 0.022)/4 = 0.242.

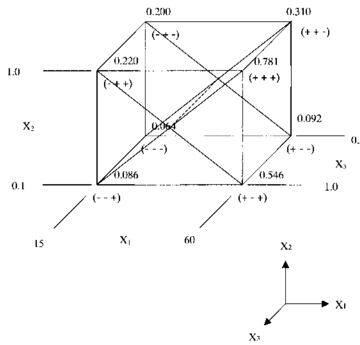


Figure 10 Determination of interaction effect of synthesis time vs. concentration of HNO_3 aqueous solution (X_1 vs. X_2) on the yield of polyaniline powder.

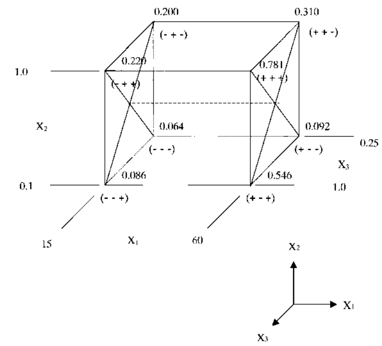


Figure 12 Determination of interaction effect of concentration of HNO_3 aqueous solution vs. mol ratio of APS/AN (X_2 vs. X_3) on the yield of polyaniline powder.

The same results will be obtained from either the effect of concentration of HNO_3 (X_2) individual comparisons or the effect of mol ratio of APS/AN (X_3) individual comparisons. As in the case of the main effects and the two factor interactions, the estimate of the three-factor interaction can be obtained from the difference between the average of vertices of (+) tetrahedron (Fig. 13) and the average of vertices of (−) tetrahedron (Fig. 14), i.e., $(0.781 + 0.092 + 0.086 + 0.20)/4 - (0.546 + 0.31 + 0.064 + 0.22)/4 = 0.005$.

Table VIII illustrates the summary of the main, two-factor interaction, and three-factor interaction effects of the yield of polyaniline powder. It shows that the sequence of the main effects on the yield of polyaniline powder in ascending order is the concentration of HNO_3 (0.181) < the mol ratio of APS/AN (0.242) < the polymerization time (0.290). This result implies that polymerization time is the most significant factor to affect the yield of polyaniline powder. In addition, the mol ratio of APS/AN affects the chemical oxidative polymerization of aniline monomer directly. Therefore, the mol ratio of APS/AN affects the yield of polyaniline powder more significantly than concentration of HNO_3 does.

The sequence of the two-factor interaction effects on the yield of polyaniline, in ascending order, is the concentration of HNO_3 vs. the mol ratio of APS/AN (0.004) < the polymerization time vs. the concentration of HNO_3 (0.046) < polymerization time vs. mol ratio of APS/AN (0.221). As mentioned above, polymerization time is the most important individual factor. Furthermore, the mol ratio of APS/AN is the second important individual factor. Therefore, the interaction effect between polymerization time and mol ratio of APS/AN is the highest in determining the yield of polyaniline powder. Similarly, the interaction effect between polymerization time and concentration of HNO_3 is second in order in determining the yield of polyaniline powder. In addition, the

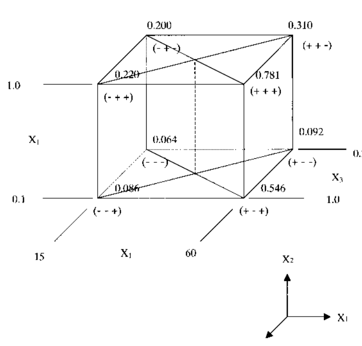


Figure 11 Determination of interaction effect of synthesis time vs. mol ratio of APS/AN (X_1 vs. X_3) on the yield of polyaniline powder.

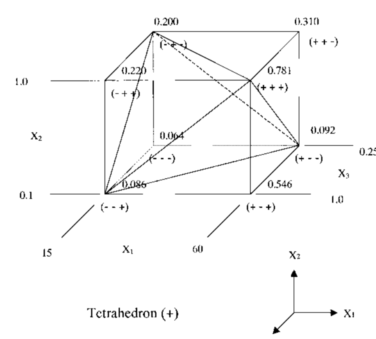


Figure 13 Determination of three-factor interaction effect of synthesis time vs. concentration of HNO_3 aqueous solution vs. mol ratio of APS/AN (X_1 vs. X_2 vs. X_3) on the yield of polyaniline powder [Tetrahedron (+)].

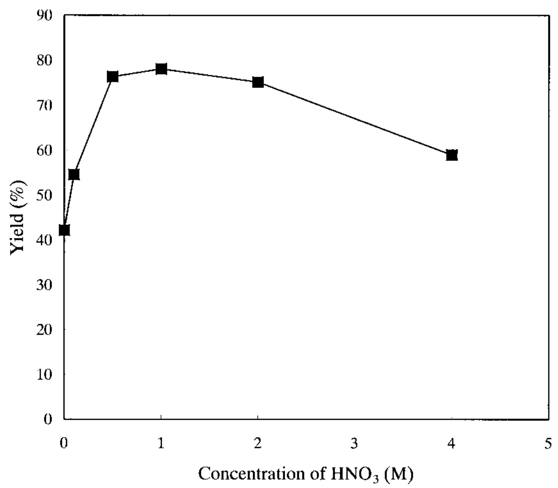


Figure 2 Influence of concentration of HNO_3 on the yield of polyaniline powder (polymerization time = 60 min, mol ratio of APS/AN = 1.0, and polymerization temperature = 0°C).

interaction effect between concentration of HNO_3 and mol ratio of APS/AN is the third in order. The three-factor interaction effect is slightly significant related to the yield of polyaniline powder. From the results of Table VIII, we can obtain a prediction equation by definition:[22]

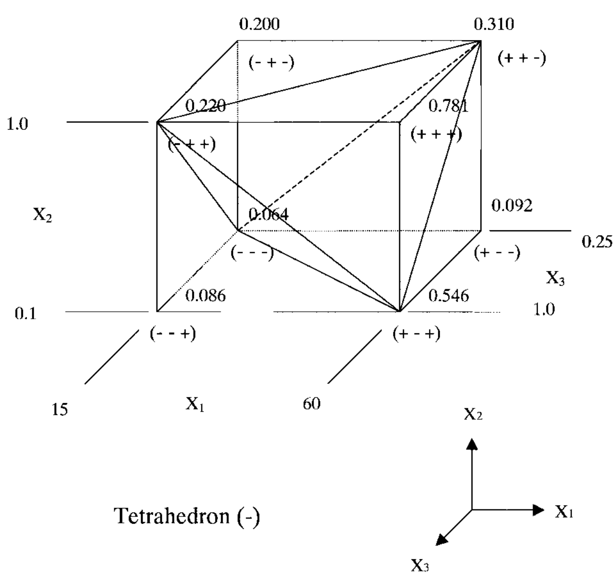


Figure 14 Determination of three-factor interaction effect of synthesis time vs. concentration of HNO_3 aqueous solution vs. mol ratio of APS/AN (X_1 vs. X_2 vs. X_3) on the yield of polyaniline powder [Tetrahedron (−)].

Table VIII Summary of Main, Two-Factor Interaction, and Three-Factor Interaction Effects of the Yield of Polyaniline Powder

Main Effect	Two-Factor Interaction Effect	Three-Factor Interaction Effect
$X_1 = 0.290$	X_1 vs. $X_2 = 0.046$	X_1 vs. X_2 vs. $X_3 = 0.005$
$X_2 = 0.181$	X_1 vs. $X_3 = 0.221$	
$X_3 = 0.242$	X_2 vs. $X_3 = 0.004$	

$$\hat{Y} = \bar{Y} + 0.145X_1 + 0.091X_2 + 0.121X_3 + 0.023X_1X_2 + 0.111X_1X_3 + 0.002X_2X_3 + 0.003X_1X_2X_3$$

where: $\hat{Y}$ is the predicted response, $\bar{Y} = 0.287$ (the average of all response values from the experimental data); X_1, X_2, X_3 = +1 (if high level) or −1 (if low level).

CONCLUSIONS

The influences of polymerization time, concentration of HNO_3, and the mol ratio of APS/AN have been illustrated. The optimum polymerization time is observed as 60 min. Results show if the polymerization time is 60 min, concentration of HNO_3 is 1.0 *M* and the mol ratio of APS/AN is 1.0, then, the yield of polyaniline powder is up to 78.1%. Moreover, the the yield of polyaniline powder increases significantly with the polymerization time, concentration of HNO_3 and the mol ratio of APS/AN.

We successfully applied a 2^3 factorial experimental design to study the main, two-factor interaction, and three-factor interaction effects of polymerization time, concentration of HNO_3, and mol ratio of APS/AN, on the yield of polyaniline powder. The sequence of the main effects on the yield of polyaniline powder, in ascending order, is concentration of HNO_3 (0.181) < mol ratio of APS/AN (0.242) < polymerization time (0.290). The sequence of the two-factor interaction effects on the yield of polyaniline powder, in ascending order, is concentration of HNO_3 vs. mol ratio of APS/AN (0.004) < polymerization time vs. concentration of HNO_3 (0.046) < polymerization time vs. mol ratio of APS/AN (0.221). The three-factor interaction effect (0.005) is slightly significant related to the yield of polyaniline powder. The prediction equation is:

$$\hat{Y} = 0.287 + 0.145X_1 + 0.091X_2 + 0.121X_3 + 0.023X_1X_2 + 0.111X_1X_3 + 0.002X_2X_3 + 0.003X_1X_2X_3$$

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