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Research article

Biopolymeric flocculant extracted from potato residues using alkaline extraction method and its application in removing coal fly ash from ash-flushing wastewater generated from coal fired power plant

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Abstract: Ash-flushing wastewater generated by coal fired power plant generally contains high massive suspended coal fly ashes, which limit the recycling of ash-flushing wastewater and contaminate the environment due to the release of heavy metals from coal fly ashes. In this study, the Biopolymeric Flocculant extracted from Potato Residues (BFPR) under alkaline condition was applied to treat ash-flushing wastewater. The extraction conditions were optimized. The results showed that the highest yield of 58.77% BFPR was extracted from potato residues under the optimum condition: temperature 100 °C, potato residue concentration 42.09 g/L, extraction time 25.27 min, NaOH concentration 0.48 M and $MgSO_4 \cdot 7H_2O$ concentration 0.4 g/L. Gel permeation chromatography analysis indicated that the approximate molecular weight (MW) of BFPR was 4781 kDa. Fourier transform infrared spectroscopy indicated the presence of carboxyl, hydroxyl, and methoxyl groups in BFPR. Furthermore, BFPR showed good flocculating efficiency of 93.44% to ash-flushing wastewater when 8.3 mg/L BFPR was added, and thus achieved the recycling of ash-flushing wastewater.

Keywords: biopolymeric flocculant; potato residues; coal fly ashes; ash-flushing wastewater

1. Introduction

The coal-firing power plant produces a huge amount of ash-flushing wastewater during cleaning the boilers, pipes and other equipments [1]. The ash-flushing wastewater is characterized by strong alkaline pH, its pH value is normally higher than 9.5 due to the accumulation of massive CaO and MgO in ash-flushing wastewater [2,3]. Moreover, the ash-flushing wastewater generally contains high concentration of suspended coal fly ashes, which are dust-like particles and limit the recycling of wastewater in ash-flushing process [4]. In addition, coal fly ashes are mainly composed by oxides of silica, aluminium, iron, calcium, magnesium and toxic heavy metal ions such as Cu, Cr, Zn, Cd and Pb [2,5-9]. Therefore, the direct discharge of ash-flushing wastewater will cause serious environmental pollution due to the release of heavy meals from coal fly ashes [5,7,10,11].

Flocculant agent is a good choice to remove the suspended coal fly ashes from ash-flushing wastewater. Inorganic flocculants such as aluminum sulfate and chemically synthetic flocculants such as polyacrylamide derivatives are toxic to humans and environment [12,13]. Moreover, inorganic flocculant are normally pH sensitive, and thus not suitable for the treatment of strong alkaline wastewater. Biopolymeric flocculants are biopolymeric substances and thus advantageous over inorganic and chemically synthetic flocculants due to their nontoxic, biodegradable properties and wide adaption to pH variation [14-16]. Biopolymeric flocculants can be produced by microorganisms during their growth [17,18], or extracted from plants [19]. However, biopolymeric flocculants produced by microorganisms require expensive fermentation medium and fermentation process [20]. To reduce the production cost of biopolymeric flocculants produced by microorganisms, various wastewaters [21-26], activity sludge [27,28] and hydrolyzates of agricultural wastes [4,29,30] were used as non-expensive carbon source to produce biopolymeric flocculants. However, the fermentation process is still necessary, which increase the production cost of biopolymeric flocculant. Considering some plant residues are rich in biopolymeric substances, such as polysaccharides, the attempts to extract biopolymeric substances from plant residues were reported. For example, pectic polysaccharides extracted from cactaceae were used as biopolymeric flocculants to treat wastewater [19]. However, the research on extraction of biopolymeric flocculants from agricultural residues is still very lack.

Potato residues are an agricultural by-product, which is generated in the industrial process of potato [31,32]. These by-products are partially integrated in animal feed or used for biogas production and extraction of pectin, but most of them are stacked outside without any disposal, which consequently cause environmental pollution and the waste of resources [32]. The potato residues generally contain biopolymeric substances, such as protein, starch, pectin, cellulose and hemicellulose [31,32]. Thus, it has potential to convert potato residues into added-value products, such as biopolymeric flocculant.

In this study, the biopolymeric flocculant was produced for the first time from potato residues using the alkaline extraction method and the condition for Biopolymeric Flocculant extracted from Potato Residues (BFPR) was optimized. Subsequently, BFPR was applied to remove coal fly ashes from ash-flushing wastewater generated from coal fired power plant. The results showed that the highest yield of 58.77% BFPR was achieved under the optimum conditions. Furthermore, BFPR exhibited high efficiency of 93.44% in removing coal fly ash from ash-flushing wastewater, and thus achieved the recycling of coal ash-flushing wastewater.

2. Materials and Method

2.1. Materials

The potato residues were collected from potato starch processing plant of Heilongjiang province, China. After natural air drying, the potato residues were crushed into powders and sieved using a 60 mesh sieve and then stored at room temperature. The chemicals used in this study were analytical grade and purchased from Sigma Chemicals Company or Sinopharm Chemical Reagent Company. The deionized water was used throughout all the experiments.

2.2. Flocculating activity assay

Flocculating activity of BFPR was determined by calculating the flocculating rate according to a previous study [33]. Briefly, kaolin clay was used as the solid phase. 60 mL kaolin suspension of 5 g/L was added with BFPR to different concentrations and the pH of the flocculation system was adjusted to pH 7.0. After stirring for 2 min and settling for 1 min, the absorbance (OD_{550}) of the supernatant was measured by a spectrophotometer (Unic-7200). A control experiment, without addition of any agent, was measured in the same manner. The flocculating rate was calculated according to the following equation: flocculating activity = $[A - B]/A \times 100\%$, where B is the absorbance of the sample at 550 nm and A is the absorbance of the control at 550 nm.

2.3. Effects of pH and temperature on the extraction of BFPR

The initial pH of potato residues suspension was adjusted using 3 M NaOH solution and 1 M HCl solution. The effects of pH variation in the range of 2.0 to 13.5 on flocculating activity and the yield of BFPR were analyzed. To determine the optimum extraction temperature, the 40 g/L potato residues suspension (pH 13) was incubated at 50, 60, 70, 80, 90 and 100 °C for 30 min, then the flocculating activity and the yield of BFPR were measured. To optimize the potato residues concentration, the flocculating activity and the yield of biopolymeric flocculant extracted from suspension containing 10, 20, 30, 40, 50, 60, 80 and 100 g/L potato residues were analyzed. To further optimize the extraction conditions, the influences of NaOH concentration varied from 0.06 to 0.6 M, the extraction time varied from 5 to 60 min, $MgSO_4 \cdot 7H_2O$ concentration varied from 0 to 1.0 g/L on the flocculating activity and the yield of BFPR were evaluated.

2.4. Experimental design and statistical analysis

Response surface methodology was further applied to determine the optimum condition for the extraction of BFPR. Based on the above experiments, three extraction parameters including potato residues concentration, extraction time and NaOH concentration were identified as key factors responsible for extraction yield. The optimization was designed based on a three-factor Box-Behnken design with a total of 17 experimental runs. The experimental runs for Box-Behnken design were listed in Table 1, potato residues concentration (35 to 45 g/L), extraction time (20 to 30 min) and NaOH concentration (0.42 to 0.54 M) were designed as A, B, C and prescribed into three levels, coded −1, 0 and +1 for low, intermediate and high value, respectively. Each experimental run was performed in three replicates. The response variable (Y), representing the extraction yield, was fitted using a second-order polynomial equation (1):

$$Y = \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_1\beta_2AB + \beta_1\beta_3AC + \beta_2\beta_3BC + \beta_1\beta_1A^2 + \beta_2\beta_2B^2 + \beta_3\beta_3C^2 \quad (1)$$

where, Y is the response (yield of BFPR); β_0 is the offset term; β_1, β_2 and β_3 are the linear coefficients; $\beta_1\beta_1$, $\beta_2\beta_2$ and $\beta_3\beta_3$ are the quadratic coefficients, and $\beta_1\beta_2$, $\beta_1\beta_3$ and $\beta_2\beta_3$ are the coefficients of the linear-by-linear interaction effect between independent variables A (potato residues concentration), B (extraction time) and C (NaOH concentration). Design-Expert software (v.8.0.6, Stat-Ease, Inc, Minneapolis, USA) was used for the experimental design and the experimental data analysis. The adequacy of the model was determined by evaluating the lack of fit, the coefficient of determination (R^2) and the F-test value obtained from the analysis of variance (ANOVA) [34].

Table 1. Box-Behnken design matrix with the observed and predicted values.

Std. No.	Run	A	B	C	Actual yield (%)	Predicted yield (%)
1	14	35	20	0.48	50.63 ± 0.81	50.77
2	3	45	20	0.48	54.90 ± 0.29	54.73
3	16	35	30	0.48	52.36 ± 0.71	52.53
4	17	45	30	0.48	55.05 ± 0.67	54.91
5	7	35	25	0.42	50.60 ± 1.03	50.26
6	1	45	25	0.42	54.77 ± 1.74	54.74
7	5	35	25	0.54	52.75 ± 0.75	52.78
8	4	45	25	0.54	54.31 ± 0.78	54.65
9	9	40	20	0.42	50.52 ± 0.50	50.72
10	15	40	30	0.42	52.36 ± 0.00	52.53
11	13	40	20	0.54	52.94 ± 1.00	52.77
12	11	40	30	0.54	53.11 ± 0.06	52.91
13	10	40	25	0.48	58.32 ± 0.37	57.76
14	8	40	25	0.48	57.74 ± 2.50	57.76
15	6	40	25	0.48	57.49 ± 0.55	57.76
16	2	40	25	0.48	57.33 ± 0.59	57.76
17	12	40	25	0.48	57.93 ± 0.28	57.76

2.5. Extraction and the yield of biopolymeric flocculant

In order to obtain BFPR, the dried potato residues were crushed into powders and sieved using a 60 mesh sieve. The potato residue powers were added into 0.48 M NaOH solution at a concentration of 42.09 g/L and 0.4 g/L $MgSO_4•7H_2O$ was added into the mixture. The extraction process was performed at 100 °C for 25.27 min. After cooling, the suspension was centrifuged at 10000 rpm for 10 min to remove the solid residues. The supernatant was collected and added with two volumes of cold absolute ethanol to precipitate BFPR. The resulting precipitate was collected by centrifugation at 10000 rpm for 5 min, washed twice using 75% ethanol, and then lyophilized to obtain BFPR. To determine the flocculating activity, the extracted BFPR was dissolved in water to get a 10 g/L BFPR solution (pH around 8.6). The yield of BFPR was calculated as the following equation:

yield (%) = A/B × 100, where A is the weight of BFPR and B is the total weight of potato residues added.

2.6. Characterization of BFPR

The molecular weight of BFPR was determined by gel permeation chromatography (GPC) using a Hitachi L-6200 system controller [29]. Functional groups in BFPR were measured using a Fourier Transform Infrared (FTIR) spectrophotometer (Bruker Tensor 27, Germany). The spectrum of the sample in the KBr pellet was recorded on the spectrophotometer over a wave-number range of 600–4000 cm^{-1}.

2.7. Ash-flushing wastewater treatment using BFPR

The ash-flushing wastewater was collected from Xuzhou power plant, Jiangsu province, China. To analyze the flocculating efficiency of BFPR to ash-flushing wastewater, 60 mL wastewater was poured into a 100 mL beaker, and added with BFPR to different concentrations (2.1, 4.2, 8.3, 12.5, 16.7, 20.8, 25.0, 33.3, 41.7, 83.3 and 166.7 mg/L). Then the solution was stirred with rapid mixing for 2 min, followed by slow mixing for 1 min. After settling for 1 min, the absorbance (OD_{550}) of the supernatant was measured using a spectrophotometer (Unic-7200). A control experiment without BFPR addition was measured in the same manner. The flocculating efficiency of ash-flushing wastewater was calculated according to the following equation: FE = [A − B]/A × 100%, where FE is the flocculating efficiency of ash-flushing wastewater; B is the turbidity (OD_{550}) of the supernatant of the flocculated wastewater and A is the turbidity of the control.

2.8. Statistical analysis

All the experiments were carried out in triplicates. Data present the average value and the standard deviation of three individual experiments.

3. Results and Discussion

3.1. The effect of pH and temperature on the yield and the flocculating activity of BFPR

The effects of pH variation from 2.0 to 13.5 on the yield and the flocculating activity of BFPR were analyzed. As shown in Figure 1A, the yield around 30% was achieved in the pH range from 2.0 to 3.0; however, less than 35% flocculating activity was observed in this pH range. The strong alkaline condition (pH over 13) could significantly improve the flocculating activity and yield of BFPR. When the pH value was 13.5, the yield of 43.3% and the flocculating activity of 92.4% were achieved. Therefore, the strong alkaline condition was selected for the extraction of BFPR. The potato residues are mainly composed of polysaccharide, such as pectin, cellulose and starch, most of which could serve as biopolymeric flocculant. Pectins are complex polysaccharides, composed mainly of a-1,4-linked D-galacturonic acid (Gal A) chains in which the carboxyl groups of the Gal A can be free or methyl-esterified [35]. In previous studies, acidic or neutral pH was selected for most pectic polysaccharides extraction from potato residues [36], lemon by-products [35], apple pomace [37] and passion fruit peel [38]. However, the yield of pectic polysaccharides is very low (only 10% to 20%).

Other polymeric substances which could serve as bioflocculant are failed to be extracted. In this study, the biopolymeric flocculant was extracted from potato residues under strong alkaline condition. The achievement of high yield and high flocculating activity under strong alkaline condition could be explained by the break of ester bond and the expose of the carboxyl groups which have been reported as main functional groups in flocculation process [15].

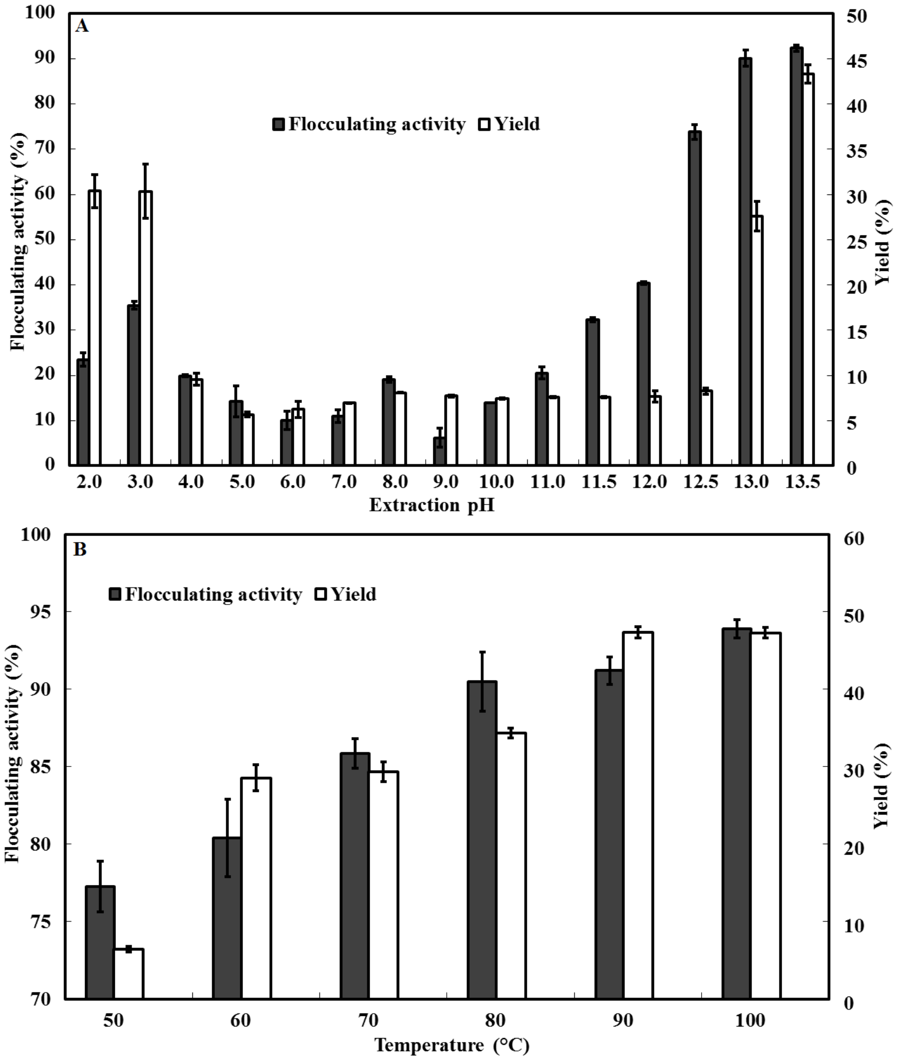

Figure 1. Effects of pH (A) and temperature (B) on the yield and flocculating activity of biopolymeric flocculant from potato residues.

The temperature could influence the extraction efficiency of natural macromolecular polysaccharides from plant residues [35,36]. As shown in Figure 1B, the flocculating activity increased from 77.3% to 94.9% and the yield increased from 6.5% to 47.3% when temperature enhanced from 50 to 100 °C. This may be due to the high temperature could accelerate the deesterification induced by high concentration of NaOH and the release of macromolecular polysaccharides into extraction solution. Therefore, the temperature of 100 °C was selected for BFPR extraction.

3.2. Effects of potato residues and NaOH concentration on the flocculating activity and the yield

The optimum concentration of potato residues would lead to the maximum extraction efficiency of BFPR. It can be seen in Figure 2A that the yield more than 40% and the flocculating activity over 85% were achieved when the concentration of potato residues was lower than 40 g/L, and the yield and the flocculating activity of BFPR decreased with the further increase of potato residues concentration. Too high concentration of potato residue would inhibit the extraction of biopolymeric flocculant. Therefore the potato residues concentration of 40 g/L was selected for further studies.

Based on the above experiment, high alkaline condition could improve the extraction efficiency of BFPR. To further enhance the yield of BFPR, the NaOH concentration was optimized. As shown in Figure 2B, the yield of BFPR increased with the increase of NaOH concentration, the highest BFPR yield of 52.3% was achieved at the NaOH concentration of 0.48 M. Excessive addition of NaOH caused the degradation of macromolecular polysaccharides and the decrease of the BFPR yield. So, NaOH concentration of 0.48 M was used in the following experiments.

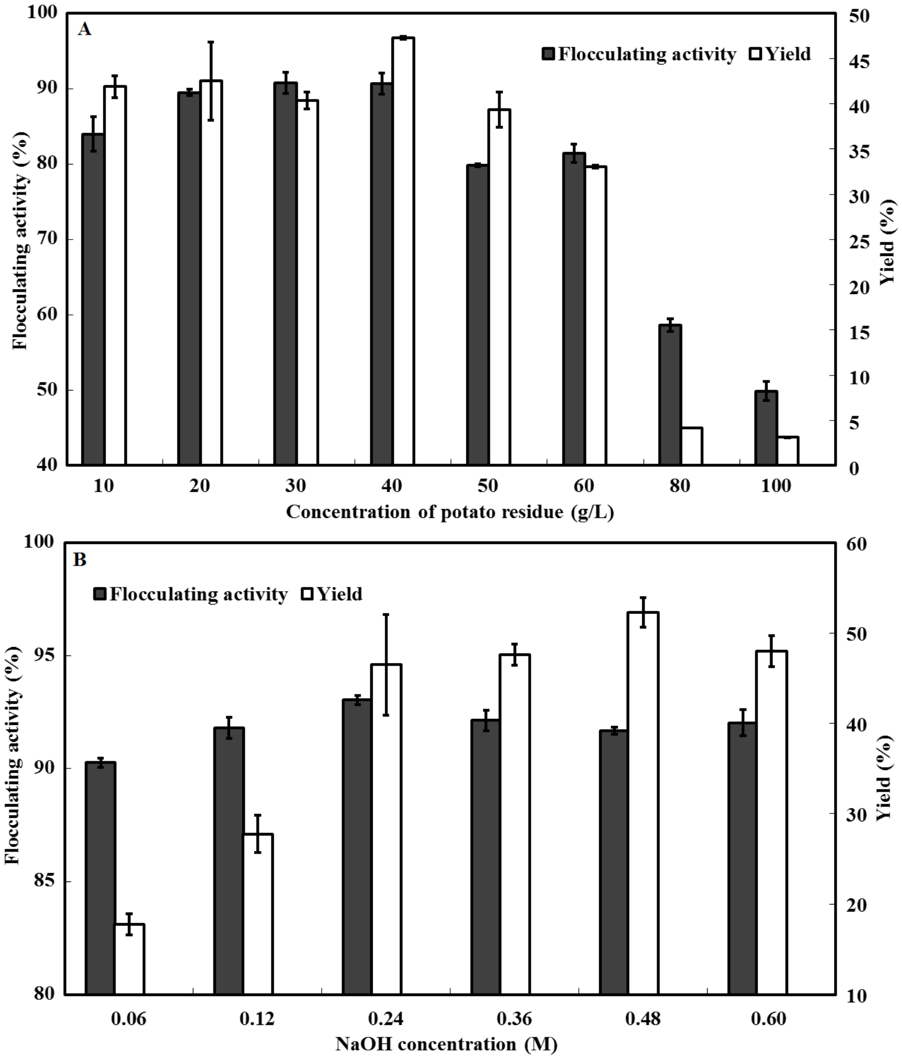

Figure 2. Effects of potato residues concentration (A) and NaOH concentration (B) on the yield and flocculating activity of biopolymeric flocculant from potato residues.

3.3. Effects of extraction time and $MgSO_4$•$7H_2O$ concentration on the flocculating activity and the yield

Figure 3A shows the influence of extraction time on the yield and the flocculating activity of BFPR. The yield increased from 42.9% to 53.3% and the flocculating activity increased from 89.30% to 94.05% when the extraction time extended from 5 to 25 min. However, too long extraction time would cause the degradation of macromolecular polysaccharides under strong alkaline condition. Further extension of extraction time resulted in the decrease of the yield and the flocculating activity.

Moreover, the preliminary experiments found that $MgSO_4$•$7H_2O$ was able to improve the yield and flocculating activity of BFPR. So the concentration of $MgSO_4$•$7H_2O$ was optimized. It can be seen in Figure 3B, the yield of 58.6% and the flocculating activity of 95.73% were achieved when 0.4 g/L $MgSO_4$•$7H_2O$ was added. The addition Mg^{2+} could bridge the biopolymeric flocculant and thus enhance the flocculating activity.

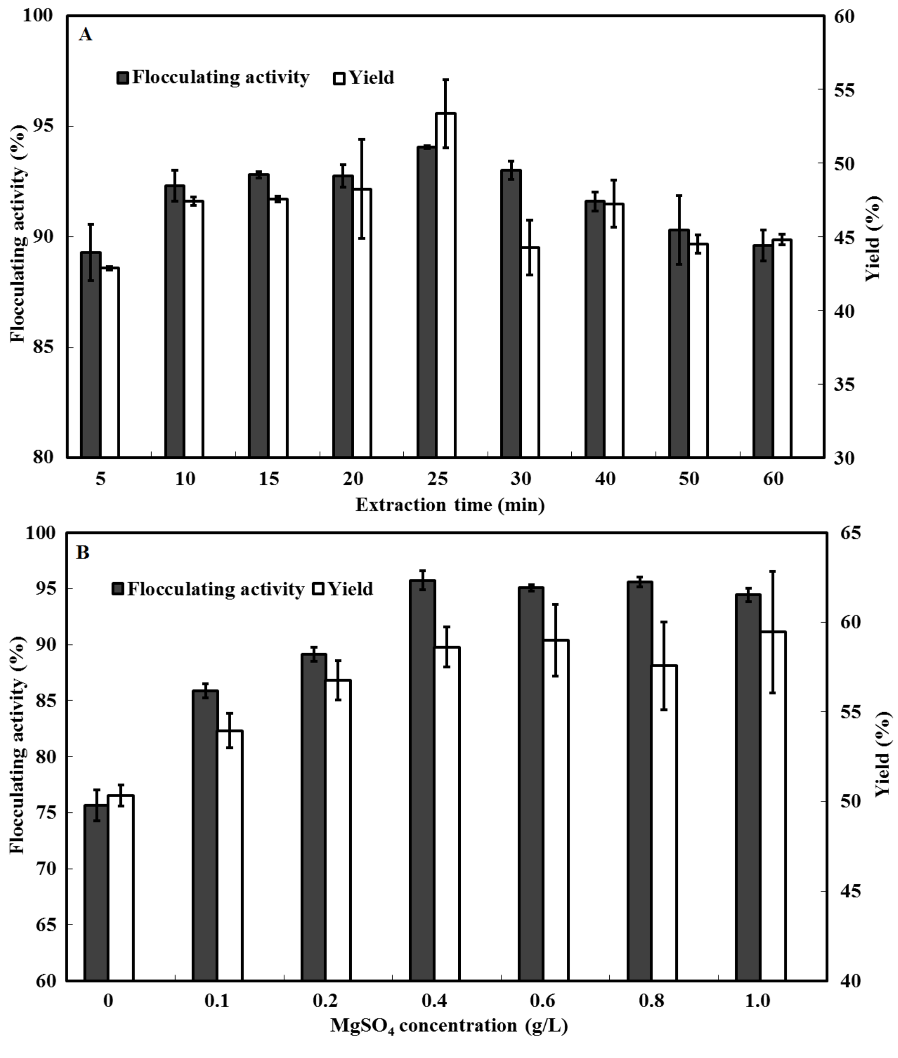

Figure 3. Effects of extraction time (A) and $MgSO_4$ concentration (B) on the yield and flocculating activity of biopolymeric flocculant from potato residues.

3.4. Statistical analysis and validation of model

According to the above one-factor-at-a-time experiments, three parameters including potato residues concentration, extraction time and NaOH concentration were identified as key factors responsible for extraction yield. All the 17 trials were conducted for optimizing these three individual parameters according to Box-Behnken design. The design matrix with the corresponding measured and predicted values was shown in Table 1. Based on the Box-Behnken design runs, a second order polynomial quadratic equation was found to be the best fit, which was expressed as follows:

$$Y = -401.892 + 7.599A + 6.799B + 886.183C - 0.016AB - 2.175AC - 1.392BC - 0.073A^2 - 0.108B^2 - 785.694C^2 \quad (2)$$

Analysis of variance (ANOVA) was used to define the adequacy of the response surface quadratic model. As shown in Table 2, the ANOVA results showed strong support for the model, with a high model F-value (84.11) and a low *p* value ($p < 0.0001$). The values of "Probability > F-value" less than 0.0500 indicated that the model terms such as A, B, C, AC, A^2, B^2 and C^2 were significant. "Lack of Fit F-value" of 1.03 for the response is non-significant which indicates that the data fit the model. Furthermore, the "pred-R^2" of 0.9280 in the design was in reasonable agreement with the "adj-R^2" of 0.9791, confirming the significance of the used quadratic model. Thus, these values indicated the adequacy of the polynomial's models accuracy and general availability.

Table 2. Analysis of variance (ANOVA) for the experimental results of the Box-Behnken design.

Source	Sum of Squares	df	Mean Square	F-Value	*p*-value Prob＞F	
Model	115.25	9	12.81	84.11	<0.0001	significant
A-PR concentration	20.13	1	20.13	132.22	<0.0001	
B-Extraction time	1.89	1	1.89	12.42	0.0097	
C-NaOH concentration	2.95	1	2.95	19.39	0.0031	
AB	0.62	1	0.62	4.10	0.0826	
AC	1.70	1	1.70	11.19	0.0123	
BC	0.70	1	0.70	4.58	0.0696	
A2	14.04	1	14.04	92.21	<0.0001	
B2	30.72	1	30.72	201.77	<0.0001	
C2	33.69	1	33.69	221.26	<0.0001	
Residual	1.07	7	0.15			
Lack of Fit	0.47	3	0.16	1.03	0.4681	not significant
Pure Error	0.60	4	0.15			
Cor Total	116.31	16				

R-squared = 0.9908, Adj R-squared = 0.9791, Pred R-squared = 0.9280, Adeq precision = 25.065.

The three dimensional response surface plots and contour plots for the yield of BFPR are represented in Figure 4. These plots illustrate the relative effects of any two factors while the third factor is kept constant at zero level. According to the numerical optimization by the software, the predicted optimum conditions were as following: the potato residues concentration 42.09 g/L, extraction time 25.27 min and NaOH concentration 0.48 M. The maximum yield of BFPR was estimated to be 58.10%, and the actual yield obtained under the predicted optimum condition was 58.77 ± 0.43% (n = 3), which is in close agreement to the modal prediction. The yield of 58.77% is similar to the yield of 58.60% before the optimization using response surface methodology, however, the concentration of potato residues enhanced from 40 to 42.09 g/L, thus the BFPR weight obtained from the same volume of extraction solution was improved.

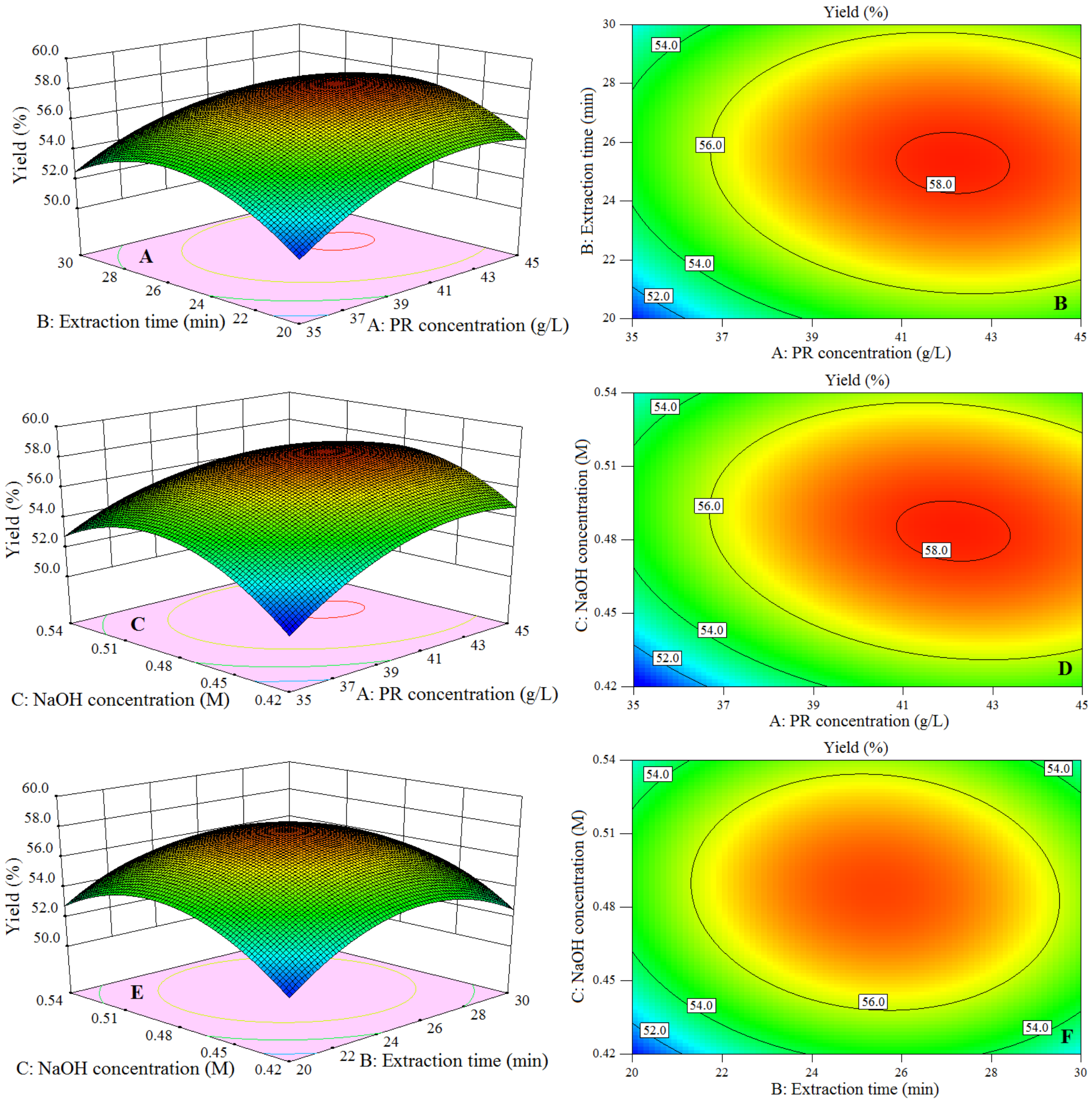

Figure 4. Three dimensional response surface and contour plots: interactive effects of (A) and (B) varied potato residue (PR) concentration and extraction time at NaOH dosage 0.48 M; (C) and (D) varied PR concentration and NaOH dosage at extraction time 25 min; (E) and (F) varied extraction time and NaOH dosage at PR concentration 40 g/L.

3.5. Characteristics of BFPR extracted under strong alkaline condition

Gel permeation chromatography result showed that the approximate molecular weight of BFPR extracted under strong alkaline condition was 4781 kDa, which is larger than that of the biopolymeric flocculants produced by different microorganisms [4,29,30,39]. FTIR spectrum was performed to reveal the functional groups of BFPR. As shown in Figure 5, the result showed a broad stretching peak in the range from 3300–3400 cm^{-1} which can be generated by stretching from hydroxyl group, and a weak peak at 2910 cm^{-1} indicated C-H asymmetrical stretching vibration and known to be typical of carbohydrate derivatives. The band at 1630 cm^{-1} displayed a carboxyl group and a weak symmetric stretching band near 1430 cm^{-1} could be attributed to the symmetric stretching of $–COO^{-}$ group, which are indicative of the presence of uronate in BFPR. The absorption in the range from 1000–1200 cm^{-1} indicated the presence of ester group, which is consistent with the fact that the biopolymeric flocculant was extracted from potato residues. In summary, FTIR spectrum of biopolymeric flocculant showed the presence of hydroxyl, carboxyl groups, which are all preferable functional groups for the flocculation process [29,40]. These negative charge groups could react with the positively charged site of suspended particles, and thus the particles can approach sufficiently close to each other so that attractive forces become effective [27].

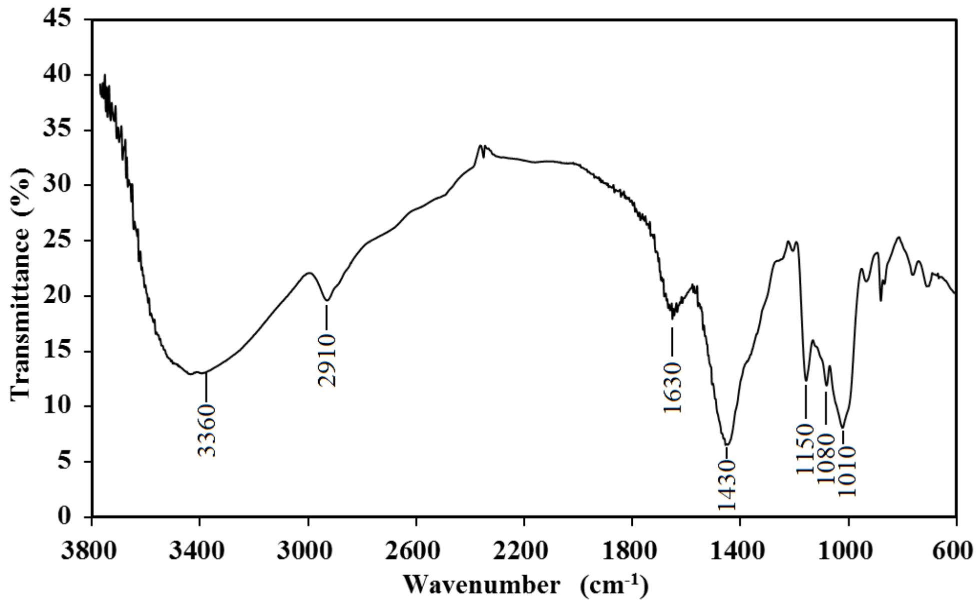

Figure 5. Infrared spectrum of biopolymeric flocculant extracted from potato residues.

3.6. Removal of coal fly ash from ash-flushing wastewater using BFPR

The ash-flushing wastewater was sampled from a coal fired power plant of Xuzhou city of Jiangsu province, China. This wastewater contains massive solid suspended particles (18.33 g/L coal fly ash), and its pH value was 9.88. The feasibility of removing coal fly ash from ash-flushing wastewater using BFPR was evaluated. Figure 6 shows the effects of BFPR dosage on the flocculating efficiency of ash-flushing wastewater. The flocculating efficiency over 90% was observed in a dosage range from 4.2–41.7 mg/L. When the preferred dosage of 8.3 mg/L was added, the highest flocculating efficiency of 93.44% was achieved. Higher or lower dosage of BFPR resulted in poorer removal efficiency. The bridging phenomena could not effectively form when dosage was lower than 4.2 mg/L. And the over-addition of negatively charged BFPR caused the repulsion of negatively charged particles and thus induced the decrease of removal efficiency of coal fly ash. Therefore, BFPR could be applied to effectively remove the suspended coal fly ashes, and thus achieve the recycling of ash-flushing wastewater in coal fired power plant.

In previous studies, several flocculation mechanisms have been proposed. For examples, Li et al. developed a universal environmental friendly method for flocculating harmful algal blooms in marine and fresh water using modified sand, and the main flocculation mechanisms were proposed as charge neutralization, bridging and sweeping mechanism [41]. Yuan et al. proposed that charge neutralization and bridging-netting mechanism is associated to the flocculation of cyanobacterial cells using coal fly ash modified chitosan. In this study, BFPR exhibited a good flocculating activity to ash-flushing wastewater [42]. In further work, the mechanisms on the flocculation of ash-flushing wastewater using BFPR will be further studied.

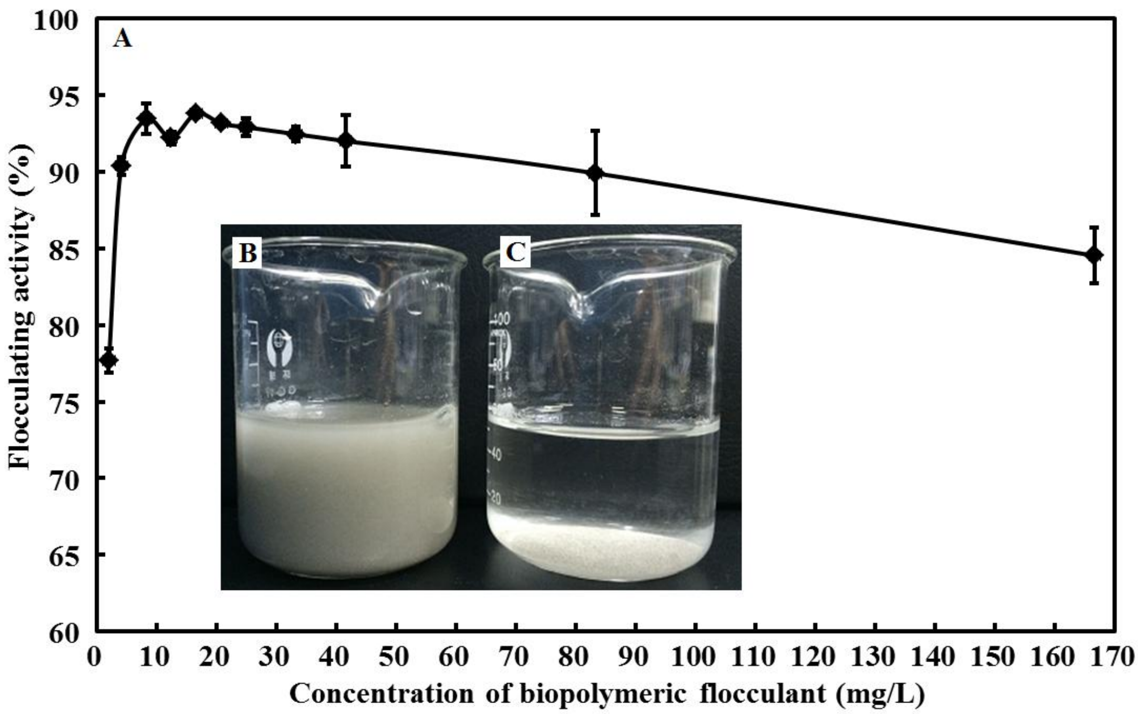

Figure 6. Application of BFPR in treating coal ash flushing wastewater. A. effect of BFPR concentration on flocculating efficiency of ash flushing wastewater; The images of ash flushing wastewater before (B) and after (C) flocculating by BFPR.

4. Conclusion

In this study, the biopolymeric flocculant was extracted for the first time from potato residues using alkaline extraction method. The highest yield of 58.77% biopolymeric flocculant was extracted from potato residues under the optimum condition: temperature 100 °C, the potato residues concentration 42.09 g/L, extraction time 25.27 min, NaOH concentration 0.48 M and $MgSO_4 \cdot 7H_2O$ concentration 0.4 g/L. The approximate molecular weight of BFPR was 4781 kDa, and its main functional groups were carboxyl and hydroxyl groups. BFPR shows good flocculating activity of 93.44% to ash-flushing wastewater when 8.3 mg/L biopolymeric flocculant was added, and thus achieved the recycling of ash-flushing wastewater.

Acknowledgments

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Conflict of Interest

All authors declare no conflicts of interest in this paper.

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