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Research Article

Research on Wheat Straw Application in the Preparation of Superplasticizer

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Wheat straw was utilized in the preparation of polycarboxylate superplasticizer (PCS) to improve its performance as well as to reduce the production cost in this paper. The addition time and pretreatment time of wheat straw in the production of PCS were detected. Orthogonal experimental design was adopted to optimize the components of reactants, and the adding time of the initiator was also examined. The PCS produced with wheat straw in this paper kept a rather high water-reducing rate. In addition, the results of some physical characteristics showed there were no obvious differences between the PCS produced with and without wheat straw, while the PCS produced with wheat straw had the longer time of coagulation. At the same time, infrared spectrum implied that the addition of wheat straw made the side chain more abundant and had little influence on the main chain of the large molecules.

1. Introduction

Polycarboxylate superplasticizer (PCS) is recognized as vital admixtures in the utilization of modern concrete technology because they could improve the workability of concrete by dispersing agglomerated hydrating cement particles present in the paste, and they could result in the highly flowable concrete or reduce the water to cement ratio of concrete [1]. There are three generations of water-reducing mixtures used in concrete. The first generation is generally manufactured from lignosulfonates and is usually called the ordinary water-reducing agent. The second one is made from sulfonated naphthalene formaldehyde or sulfonated melamine formaldehyde which belongs to the superplasticizer. Polycarboxylate superplasticizer (PCS) was introduced as the third and latest generation of concrete admixtures in the 1980s which definitely has the superior performances [2, 3]. The ordinary water-reducing lignosulfonate-based mixture has the low production cost and excellent retarding effect. However, if their additive dosages exceeded the recommended dosages in concrete, it would result in the noncondensation of concrete for several days and delay the project. And under the limited recommended dosages, the water-reducing rate is just between 6% and 12%, which cannot meet the construction requirement. The second generation of superplasticizers has better performance and acceptable production cost; however, formaldehydes are used in large quantities in their production processes, which lead to the serious environmental pollutions. The latest polycarboxylate superplasticizer (PCS) could satisfy both the construction and environment requirements; however, the production costs are pretty high [4, 5]. With the development of sustainable strategy around the world, green chemistry has become a core part of the international chemistry discipline. From the perspective of material process engineering, which is the full utilization of renewable resources, it is imperative to develop new water-reducing agents that are environmentally friendly, meet the standard of performance, and have economic feasibility [6, 7].

Cellulosic biomass as the primary product of photosynthesis exists widely in terrestrial environments, and there are a large number of active groups such as hydroxyl groups consisted in the polymer structure of cellulose, which could easily occur derivatization through esterification and etherification reaction under certain conditions and at the same time, some water-soluble cellulose derivatives show some prominent properties such as thickening, dispersion, emulsification, solubilization, and protective colloid. This derivative process has the features of extensive raw material sources and is environmental friendly, so it is widely used in many areas [8, 9]. In the field of building materials, research about cellulose derivatives such as water-reducing agent has also been attracted the attention and it has been reported that the main raw materials for the preparation of the water-reducing agent is based on biomass including cellulose, hemicelluloses, and starch and the modification methods including carboxymethylated, carboxyethyl, alkyl sulfonic acid, and sulfuric acid esterification. For example, the researchers from the United States and Japan used starch as the raw material to produce the polysaccharide water-reducing agent [10, 11]. In China, besides starch, there are researches utilizing cotton fiber, cotton linter, and bagasse as raw materials for the production of the water-reducing agent. These measures could reduce the production cost as well as reinforce the performance of concrete [12–14]. These researches show that, for all the polysaccharides such as starch, cellulose, and so on, through rational molecular design and the appropriate reaction conditions, they are probably prepared into water-reducing agents with high efficiency in concrete [15–18]. However, the systematic preparation process of the cellulose-based water-reducing agent is rarely reported.

Straws and stalks are the main part of crops, and a large number of straw and stalk wastes would be generated every year. If they are not handled properly, the serious resource wastes and environmental pollution would happen. The utilization of crop residues such as straws and stalks has been as a major strategic industry in the area of the renewable resources development in the 21st century of the world. And the resources' utilization is mainly focused on the development of chemical industry and building materials [19–21]. According to current reports, the raw materials used for the production of the water-reducing agent are mostly cellulose products. At the same time, the research on the utilization of straws and stalks directly to prepare the water-reducing agent has not been reported yet.

In this paper, wheat straw waste was joined in the preparation of polycarboxylate superplasticizer (PCS), through the graft copolymerization of the large molecules of straw fiber and PCS to improve the performance of the water-reducing agent as well as to reduce its production cost. The proper addition time of wheat straw was obtained. And the orthogonal experiment was utilized to optimize every ingredient ratio. At the same time, the addition speed of the initiator was also examined. The differences between the application performances and production cost of the PCS with and without the modification of wheat straw were compared. In addition, an infrared analysis was made to distinguish the mechanism.

2. Materials and Methods

2.1. Materials. Wheat straw was collected from the suburb of Zhengzhou, China. Before using, it was washed and soaked with deionized water and then fried at 80°C for 24 h. After that, they were ground and passed through an 80-mesh screen. Part of the sieved wheat straw was immersed in the solution of NaOH for 24 h, and the product was washed with 1% HCl and deionized water until the eluent reached neutral followed by drying at 80°C for 24 h by passing through the 80-mesh screen again. The final product was stored in a desiccator for further use in all the experiments.

Methyl allyl polyoxyethylene (TPEG) 2400 and 1000, schistose and waxy white solids, respectively, of industrial grade, were supplied by Jiangsu Haian Petroleum Chemical Factory. Maleic anhydride (MAN), flaky, white solid, was of analytical grade and purchased from Tianjin Kemiou Chemical Reagent Co., Ltd. Sodium methallyl sulfonate (SMAS), crystalline powder, was purchased from Shanghai Macklin Company. Acrylamide (AM), white solid, was of analytical grade and purchased from Tianjin Kemiou Chemical Reagent Co., Ltd. Ammonium persulfate, white, crystalline solid, was of analytical grade and also purchased from Tianjin Kemiou Chemical Reagent Co., Ltd. Sodium hydroxide, white, uniform grain, was purchased from Yantai Shuangshuang Chemical Industry Co., Ltd. The cement P.O. 42.5 was supplied by China United Cement Zhengzhou Co., Ltd.

2.2. Synthesis of PCS. TPEG-1000, TPEG-2400, maleic anhydride (MAN), sodium methallyl sulfonate (MAS), acrylamide (AM), and distilled water were added according to some proportion into a 500 ml four-neck round-bottom flask with a stirrer, a water separator with a condenser and a thermometer. The device was kept in a constant temperature water bath at a constant temperature of 80°C. When the reactants dissolved and reached the temperature of 80°C, the initiator was added through the constant pressure drop funnel. Then, the system was kept in the same temperature for several hours. When the reaction finished, it was naturally cooled to room temperature, followed by adjusting pH to 7.0 using sodium hydroxide solution. Finally, the water-reducing agent products of brown transparent liquid was obtained.

2.3. Performance Measurements. The cement fluidity of the water-reducing agent products was tested by the standard test method of the concrete additive uniformity test of China (GB/T 8077-2000), and the water cement ratio was 0.29.

The water-reducing rate determination method was according to the concrete admixture standard of China (GB8076-2008), and the calculation formula is as follows:

$$W_R = \left[\frac{(W_0 - W_1)}{W_0}\right] \times 100\%, \tag{1}$$

where W_R (%) is the water-reducing rate, W_0 (kg/m^3) is the water consumption of the benchmark concrete unit, and W_1 (kg/m^3) is the water consumption of the mineral admixture concrete unit.

Solid content determination and concrete slump tests were according to the concrete admixture standards of China (GB8076-2008); the errors were kept no more than 0.3%, and the calculation formula is as follows:

$$X_s = \left[\frac{(M_2 - M_0)}{(M_1 - M_0)}\right] \times 100\%, \quad (2)$$

where X_s (%) represents the solid content, M_0 (g) is the quality of the weighing bottle, and M_1 (g) is the quality of the original sample, while M_2 (g) is the quality of the weighing bottle and the dried sample.

In the determination of compressive strength, all the processes such as concrete mixing, molding, measurement, and data processing were in accordance with the ordinary concrete mechanics performance standard test method of China (GB/T50081-2002). The concrete was prepared according to the concrete admixture standard of China (GB/8076-2008), where the specified ratio of C (cement) : S (stone) : G (sand) : W (water) in the concrete mixture was 1 : 2.11 : 3.5 : 0.48 and the cement type was P.O 42.5 R, which was purchased from China United Cement Group Co., Ltd, in Henan Dengfeng. The sand and stone were produced in Jiayu town, Zhengzhou, Henan Province, China.

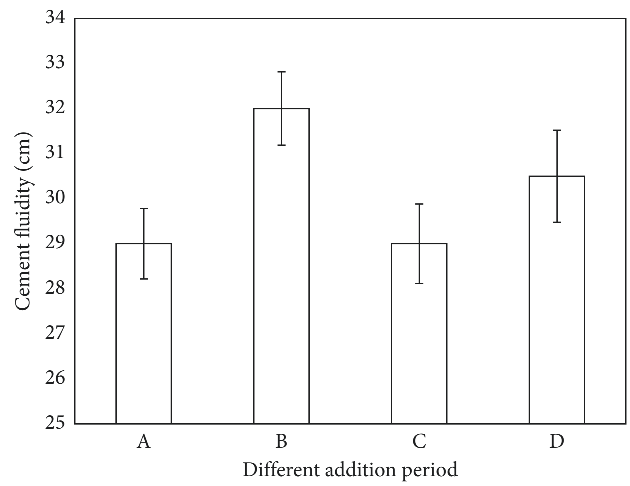

Figure 1: Effect of different addition periods of wheat straw (WS) on cement fluidity. A: no WS, B: WS added initially, C: WS added with the initiator, D: WS added after the initiator.

3. Results and Discussion

3.1. Addition of Wheat Straw in the Production of PCS

3.1.1. Effect of Different Addition Periods of Wheat Straw on Cement Fluidity. A mature technology of the preparation of PCS was selected through experimental comparison. The effect of different addition periods of wheat straw on cement fluidity was firstly examined, and the result is shown in Figure 1, where A represents the original process without wheat straw (WS), B represents that the wheat straw (WS) was initially added with other reactants, C represents that the wheat straw (WS) was added with the initiator, and D represents that the wheat straw (WS) was added after the addition of the initiator. It is clearly seen that the cement fluidity increased along with the addition of wheat straw, and the results implied that the performance of PCS could be improved through the addition of wheat straw. It could be seen that the maximum cement fluidity was obtained when the wheat straw was joined at the beginning of the reactions, and the cement fluidity increased a little when the wheat straw was added after the addition of the initiator. However, there was no change observed when the wheat straw added with the initiator. The results meant that wheat straw improved the fluidity performance of PCS maybe through graft copolymerization to the main chain macromolecules, and the mechanism should not be the free-radical polymerization, because the radical initiator could inactivate the effect of wheat straw. The better and feasible way was to add the wheat straw in the beginning with other reagents.

3.1.2. Effect of Pretreatment Time of Wheat Straw on Cement Fluidity. As is well known, wheat straw is composed of macromolecules such as cellulose, hemicelluloses, and lignin. In order to make the wheat straw to react sufficiently, pretreatment of wheat straw was utilized. On the basis of references and work experiences, hydrochloric acid with the concentration of 0.5 mol/L was selected to immerse wheat straw, and then, the mixture was kept at room temperature for different periods of time; after that, the mixture was suction filtered through the Büchner funnel and rinsed with distilled water at the same time in order to reduce the acidity of wheat straw; finally, it was dried and reserved for use. Figure 2 shows the effect of different pretreatment time periods of wheat straw on cement fluidity, where the pretreatment wheat straw of different time periods was added in the same quality at the beginning with other reactants. It can be seen from Figure 2 that when the pretreatment time of wheat straw was 0 and 12 h, the cement fluidity remained around 32 cm. However, when the pretreatment time increased to 18 h, the cement fluidity decreased a little. When the pretreatment time continued to extend, the cement fluidity decreased more. The results implied that the pretreatment was not in favor of the reactions because the improvement of the fluidity of PCS produced with wheat straw should be attributed to the graft copolymerization of

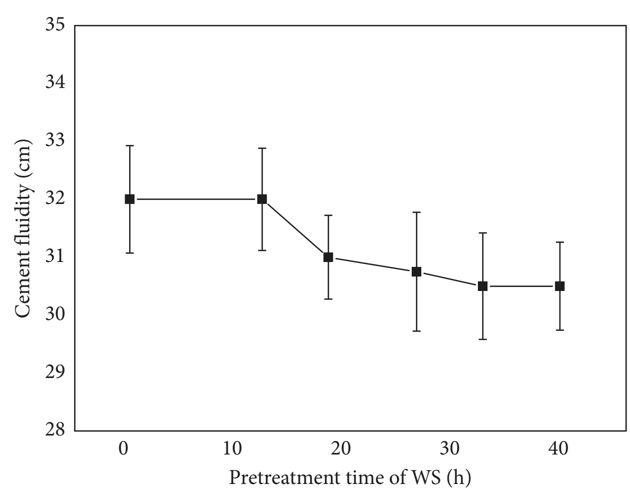

Figure 2: Effect of different pretreatment time periods of wheat straw (WS) on cement fluidity.

Table 1: Orthogonal experimental design for nine trials with three levels of concentrations for each reactant components' variable and corresponding cement fluidity.

Run	TPEG-2400	TPEG-1000	MAS	WS	Cement fluidity (cm)
1	1	1	1	1	27.5
2	1	2	2	2	28.75
3	1	3	3	3	29.75
4	2	1	2	3	31
5	2	2	3	1	28.25
6	2	3	1	2	31.5
7	3	1	3	2	32.25
8	3	2	1	3	30.75
9	3	3	2	1	32
MV 1	28.67	30.25	29.92	29.29	—
MV 2	30.25	29.25	30.92	30.83	—
MV 3	31.67	31.08	30.08	30.5	—
EV	3	1.83	1	1.58	—

Table 2: Levels of variables used in the experimental design.

Variable	Low level (1)	Middle level (2)	High level (3)
TPEG-2400 (g)	15	22.5	30
TPEG-1000 (g)	6.25	12.5	18.75
MAS (g)	1	2	3
WS (g)	1	2	3

the large molecules of wheat straw, while the pretreatment reduced the molecular weight of the wheat straw, which resulted in the decrease of the fluidity. So in the following experiments, the wheat straw without pretreatment was used directly in the reactions.

3.2. Optimization of Reactions for PCS Production with Wheat Straw

3.2.1. Optimization of Reactants for PCS Production. Orthogonal experimental design was adopted to find the optimal reactant components. Four reactants such as TPEG-2400, TPEG-1000, MAS, and wheat straw were chosen to optimize the components because they were the main ingredients that affect the product performance as well as the cost [22, 23]. Table 1 describes the orthogonal experimental design for nine trials with three levels of concentrations for each reactant components' variable and corresponding cement fluidity. At the same time, the mean values (MVs) and orthogonal extreme values (EVs) are also shown in Table 1, while Table 2 illustrates the levels of the four factors used in the experimental design. It could be seen from Table 1 that all the four factors influenced the cement fluidity of PCS directly, and the orthogonal extreme values of TPEG-2400, TPEG-1000, MAS, and wheat straw (WS) were 3, 1.83, 1, and 1.58, respectively. So, the sequence according to the effects of reactant on the cement fluidity of PCS was TPEG-2400, TPEG-1000, wheat straw (WS), and MAS. The results also verified that the performance of PCS depended on the amounts of macromolecules. In addition, it could be found that, in the orthogonal experiment design, the effect trends of TPEG-2400 and TPEG-1000 on the cement fluidity of PCS are still rising, which meant that if their dosages increased continually, the cement fluidity of PCS could improve; however, the product cost would also increase. In order to satisfy both the performance and the cost, the dosages of TPEG-2400 and TPEG-1000 were chosen to be 30 g and 18.75 g, respectively. And when the dosage for both MAS and wheat straw was 2 g, the cement fluidity of PCS had the optimal values.

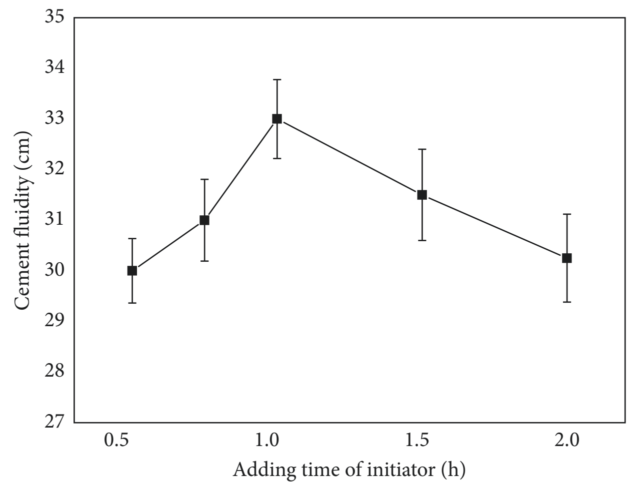

Figure 3: Effect of different adding time periods of the initiator on cement fluidity.

3.2.2. Optimization of Reaction Time for PCS Production with Wheat Straw. Considering the reaction characteristics of slow initiate, fast growth, and speed termination for free-radical polymerization, the adding time of the initiator is vital for the reactions. Temperature has a great influence on the decomposition of the initiator, and the higher the temperature is, the faster the initiator spreads; at the same time, more free radicals will be produced, and the polymerization speed is accelerated. Hence, the initiator is usually added when the reaction temperature gets the proper point, which is beneficial to the polymerization reaction. In this experiment, the proper temperature for the initiator added was 80°C, and the adding time was examined in order to obtain the optimal reaction condition. Figure 3 shows the effect of different adding time periods of the initiator on cement fluidity. The adding time was set as 0.5 h, 0.75 h, 1 h, 1.5 h, and 2 h, respectively, which meant the different adding speed of the initiator into the reaction system. It is clearly seen that the cement fluidity appeared in the parabolic trend along with the increase of adding time, and when the adding time was 1h, the maximal cement fluidity was obtained, which showed the optimal reaction condition, so the best adding time of the initiator was 1 h for this experiment.

3.3. Characteristics of the PCS Produced with Wheat Straw

3.3.1. Some Physical Properties. All the PCS produced with wheat straw appeared brown and without obvious odor with the pH around 7.0. In addition, some physical properties were also detected to better understand the characteristics of PCS produced with wheat straw in this experiment. Table 3

Table 3: Changes of density, viscosity, and solid content along with the variation in cement fluidity using the PCS produced with wheat straw.

Cement fluidity (cm)	ρ (g/ml)	μ (MPa·s)	Solid content (%)
27.5	1.182	62.3	39.2
28.75	1.190	60.0	39.5
31.5	1.193	61.6	40.2
32.25	1.192	66.8	40.5
33	1.195	65.7	40.7

shows the changes of solid content, density, and viscosity along with the variation in cement fluidity. It is clearly seen that both the solid content and density rose along with the increase of cement fluidity. At the same time, the viscosity also improved when wheat straw was utilized in the production of PCS. The viscosity of PCS without wheat straw was around 50 MPa·s, while the values of PCS with wheat straw were between 60 and 67 MPa·s. It was known that viscosity is related to the degree of molecular polymerization to some extent, so the molecular mass of PCS with wheat straw was bigger than the one without wheat straw. However, it could be seen from the table that the viscosity was not much related with the cement fluidity because the values were close and had no obvious rules.

3.3.2. Cement Fluidity under Different Dosages of PCS Produced with Wheat Straw. It is well known that the dosages of PCS have great effects on cement fluidity, so the dosages of PCS with wheat straw were tested to find the optimal dosage. Figure 4 exhibits the values of cement fluidity under different dosages of PCS produced with wheat straw. The obvious difference was observed with and without PCS, and along with the increase of dosages, the cement fluidity showed significant improvements. When the dosage was 0.5%, the cement fluidity reached the highest value, and when the dosage rose continually, the cement fluidity had no much changes. Considering the product cost, the dosage of 0.5% was chosen as the optimal one.

3.3.3. Comparison of Concrete Slump and Compressive Strength of PCS Produced with and without Wheat Straw. The concrete slump mainly refers to whether the mixing concrete is easy to be used in the construction for the workers and whether the concrete is uniformly dense. The concrete slump also reflects the properties of the concrete's fluidity, cohesion, and water retention, and all of these characteristics of concrete are affected by the performance of the water-reducing agent, so the concrete slump could be used to validate the performance of the water-reducing agent. When the concrete slump is smaller and the compressive strength is bigger, the performance of the water-reducing agent is better. Table 4 lists out the values of water-reducing rate, concrete slump, and compressive strength of the PCS samples produced with and without wheat straw. The results showed that there were no obvious differences between the two technologies, and the utilization of wheat straw in the production of PCS could maintain the high performance of PCS. And in the experiments, it was found that the PCS produced with wheat straw had longer time of coagulation, which is more beneficial for the construction. At the same time, the production costs were compared between the two technologies, and the production cost of the new technology could be reduced around 2 dollars per kilogram compared with the one without wheat straw.

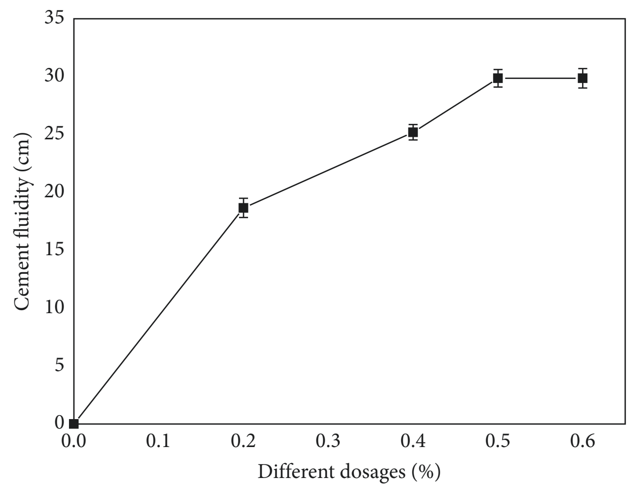

Figure 4: Cement fluidity under different dosages of PCS produced with wheat straw.

Table 4: Values of water-reducing rate, concrete slump, and compressive strength of PCS samples produced with and without wheat straw (WS).

PCS sample	Water-reducing rate (%)	Concrete slump (mm)	Compressive strength (MPa)		
			3d	7d	28d
Without WS	28.1	195	35.8	41.2	53.7
With WS	29.6	180	36.9	41.9	54.8

3.3.4. Infrared Spectrum Analysis. In order to obtain a deeper cognition of the new technology, infrared spectrum was adopted. The infrared spectrograms of PCS produced with and without wheat straw are plotted in Figures 5(a) and 5(b). It could be seen clearly that there were nine obvious peaks such as 3422.86 cm^{-1}, 2923.32 cm^{-1}, 2126.73 cm^{-1}, 1650.97 cm^{-1}, 1455.28 cm^{-1}, 1351.55 cm^{-1}, 1252.96 cm^{-1}, 1097.84 cm^{-1}, and 960.06 cm^{-1} in Figure 5(b), while there were just five obvious peaks such as 3383.15 cm^{-1}, 2110.79 cm^{-1}, 1642.52 cm^{-1}, 1350.78 cm^{-1}, and 1083.60 cm^{-1} in Figure 5(a).

These peaks indicated there were carboxyl and sulfonic groups and ether bond existed in the two products, while the hydroxyl group peak, carbon-hydrogen bond bending vibration peak, and stretching vibration peak of carbon-carbon bonds were not obvious in the product produced with wheat straw, which could be speculated that there were reactions between functional groups in wheat straw and hydroxyl group on the original chain; at the same time, the grafting reaction happened on the side chain restricted the vibration of the main chain.

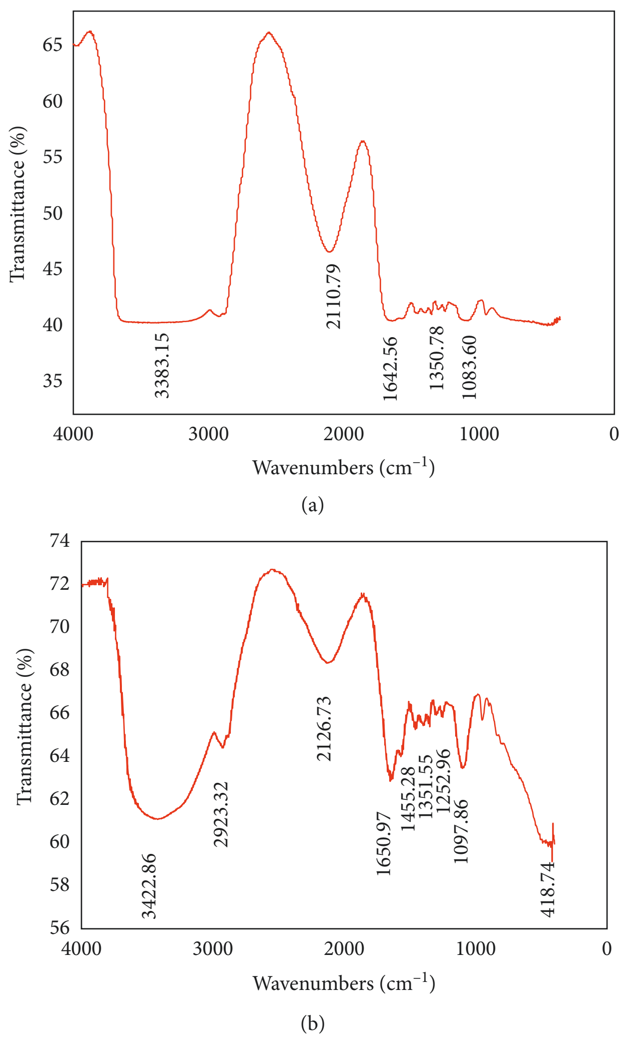

Figure 5: Infrared spectrograms of PCS produced (a) with wheat straw and (b) without wheat straw.

4. Conclusion

Experiment results showed that the utilization of wheat straw in the preparation of PCS could reduce its production cost as well as improve the performance. The optimal experimental conditions were that the wheat straw without additional chemical pretreatment was added with other reagents in the beginning of the reactions, and the mole ratios of methallyl polyoxyethylene (TPEG) 1000: methallyl polyoxyethylene (TPEG) 2400: maleic anhydride (MAN): acrylamide (AM): sodium methallyl sulfonate (SMAS) were 3 : 2 : 12 : 7 : 2. Meanwhile, the quality ratio of added wheat straw to methallyl polyoxyethylene (TPEG) 2400 was 1 : 15, and the initiator of ammonium sulfate dosage was 0.3% of the total mass fraction of monomer. The initiator concentration was 40% while the initiator adding time was 1 h, and the reaction system was kept at 80°C for 4 h at last. The rather high cement fluidity of 33 cm and the longer time of coagulation were obtained, which is more beneficial for the construction. At the same time, the production cost could be reduced around 2 dollars per kilogram product when using wheat straw. In addition, infrared spectrum revealed that the wheat straw could be grafted on to the main chain. The following research should focus on designing more rational reactions in view of molecular structure to fully develop the potential of wheat straw in the preparation of high-efficient water-reducing agents.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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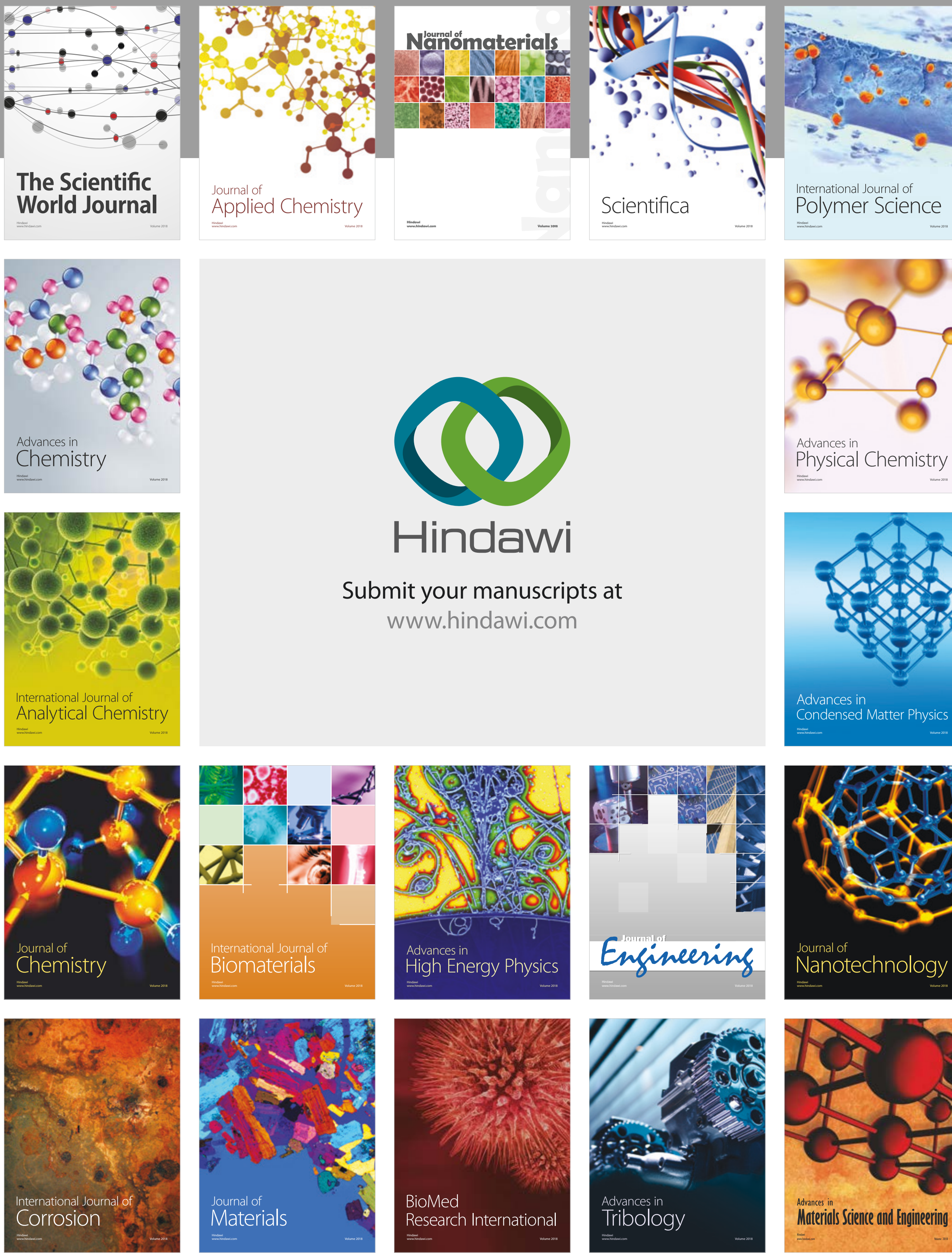

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