Optimization of effective parameters in the extraction of cellulose from sugarcane bagasse by Taguchi method and synthesis of cellulose nanostructures

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Abstract
Cellulose is one of the most important natural biopolymers and as an inexhaustible raw material can be mentioned as an industrial scale biocompatible material. Cellulose can be applied in the form of wood and plant fibers as well as energy source and building materials and ec. By producing cellulose in cost-effective and environmentally friendly ways, a nanotechnology-based product can be produced with unique and particular performance. Therefore, it is expected that these products will be replaced with petrochemical products. Nanocellulose is made from lignocellulosic primary materials. In the primary years of the current century, a great deal of research in the subject of nanocellulosic material production and application have been started and going to be global as soon as possible. In the present study, the effective parameters on cellulose extraction from sugarcane bagasse were optimized using the Taguchi statistical method. The influencing parameters in the process of cellulose consists of delignification pH, dewaxation solvents ratio, Sodium chlorite concentration and pH of hemicellulose removal. An orthogonal array L9 has been chosen for parameter design. The structure of the extracted cellulose was confirmed by XRD and IR analyzes. It is noteworthy that the structure of the synthesized nanocellulose by application of FE SEM analyses was confirmed. Optimization of the cellulose extraction process by the Taguchi method showed that importance of parameters on cellulose extraction were pH of hemicellulose removal, Sodium chlorite concentration, delignification pH, and dewaxation Solvent Ratio respectively .
Under optimum conditions at selected levels of these factors, the maximum percentage of cellulose extraction can be reached(96.667%). To evaluate the optimization process, the optimal conditions were performed in experimental form with three replication. In this condition the averaged cellulose extraction amount was 95.68%.

Keywords: Taguchi, NanoCellulose, optimization

Introduction
Cellulose is the main constituent of the cell wall, having the general formula ($C_6H_{10}O_5$) n[1]. Cellulose as an high-density polymer can be mentioned as major component of plants cell walls and due to this fact plant waste can be considered as main source of nano cellulose production. These resources include maize, rice straw, banana stems, sugar beet, soybean shell and etc[2]. The plants contain crytaline cellulose Microfibril which has been consist of 30 to 40 cellulosic linear chains[3]. Cellulose nanoparticles consist of nano-sized cellulose fibers that typically have a theoretical dimension of 5–20 nm and long dimensions varies from tens of nanometers

to several microns. The cellulose nanoparticles have a very viscous appearance and form a gel-like, translucent strip[4]. Nano sized cellulose which have been produced from acid hydrolysis has unique properties such as low density, biodegradation, and good mechanical qualities. Also, the nanocellulose is easily modified and has a high surface area and normal morphology[5]. Cellulose has a variety of properties, including high elasticity, water absorption without moisture, and heat absorber. These properties have made cellulose directly appliable in the manufacture of paints, textiles, paper. Also its derivatives can be used in medicine (cellulose acetate phthalates), military industries (Nitrocellulose), cellulose, energy drinks, synthetic sugar, alcohol and etc[3,6]. Cellulosic nano-fibers as valuable bio-natural materials are obtained mechanically by various pre-treatments of woody and non-woody plant fibers. Various enzymatic, chemical, and oxidiation pretreatments have been suggested to reduce energy consumption and further fibrillation of nanofibers[7]. Sugar cane bagasse is an agricultural waste that is produced in large quantities in sugar industry. Bagasse generally contains 40-45% cellulose, and 25-35% hemicellulose, comprising a group of polysaccharides commonly referred to as arabinose, glucose, galactose, and mannose. Other ingredients in bagasse include small amounts of lignin and minerals, waxes and other compounds[8]. Various parameters are effective in the process of cellulose extraction. By controlling and modifying these parameters simultaneously, higher yields of the desired product can be achieved. The simultaneous impact of these factors is very complex and controversial. This requires a lot of experimentation that requires time and cost. In this study, the Taguchi method was used to optimize the effective parameters in cellulose extraction and to estimate the results under optimum conditions. In the Taguchi method, the S / N ratio can be used to express the effectiveness of each parameter, where S is called the controllable parameter. However, in the experiments, there is a set of uncontrollable factors or parameters that are not considered in the design of the experiment and affect the final physical properties of the product. These parameters are called perturbation parameters and their effect on perturbation or noise[9].

Experimental

Bagasse was prepared from Ahwaz Sugar Cane Development Company. At the cellulose extraction stage, the sample was first placed in the oven for 1 day at 55 ° C. For dewaxation purposes the sample have been mixed with a specific ratio of ethanol and hexane solvents. The sample was proteolysid by adding some protease enzyme and adjusting pH = 5 in a warm water bath for a while. Then, for Delignification, the sample was added to a certain proportion of sodium chlorite and acetic acid, and lignin was removed at the specified temperature and pH. To remove hemicellulose the pH has been adjusted and properly mixed at room temperature .After the removal of hemicellulose, the sample was thoroughly washed with warm distilled water and filtrated. The extracted cellulose has been dried at 40 ° C for one day. Several factors influence the extraction of cellulose from sugarcane bagasse. Taguchi's mathematical and statistical model was applied to optimize the effective parameters in cellulose extraction. The L9 array was used in the present study. The percentage of extracted cellulose related to different extraction condition have been presented in Table 2 .Effective parameters and their levels were determined and tested by library studies [10,11,8]. Table 1 presents the effective parameters for cellulose extraction and their levels. Table 2 shows the orthogonal array used and the percentage of cellulose extracted.

Table 1. The levels of the independent operation variables

row	parameters	Levels		
1	Solvent ratio in dewaxing process (ethanol/Hexane)	0.1	0.5	0.9
2	Sodium chlorite concentration	0.3	0.6	0.9
3	pH of Delignification	4	6	8
4	pH of Hemicellulose removal	9	11	13

Table2. Taguchi experiment matrix and corresponding experimental results

raw	Solvent ratio	Sodium chlorite concentration	pH of Delignification	pH of Hemicellulose removal	%Extraction
1	0.9	0.9	8	13	75
2	0.9	0.6	6	11	94
3	0.9	0.3	4	9	82
4	0.5	0.6	8	9	87
5	0.5	0.3	6	13	76
6	0.5	0.9	4	11	96
7	0.1	0.3	8	11	85
8	0.1	0.9	6	9	88
9	0.1	0.6	4	13	78

Acid hydrolysis was used to synthesize cellulose nanostructures[12].

Results and discussion

The results of Taguchi's statistical analysis based on signal to noise ratio are shown in Table 3 and Figure 1. The highest S / N values for each variable represent the optimal test conditions. The higher the signal-to-noise ratio, the better that is for surfaces with higher signal-to-noise ratios; the more appropriate and optimal the test mode for cellulose extraction from sugarcane bagasse[13]. Delta represents the signal-to-noise ratio changes in the expriment. Using Rank we find the effect of the parameters; Rank = 1 is the parameter that has the most effect on the cellulose extraction process. Finally, sodium chlorite concentration with rank = 2 and Delta = 5.33 is the second most effective parameter in the cellulose extraction process.

Table 3: Response Table for Means

Level	solvent ratio	pH of Delignification	Sodium chlorite concentration	pH of Hemicellulose removal
1	83.67	85.33	81.00	85.67
2	86.33	86.00	86.33	91.67
3	83.67	82.33	86.33	76.33
Delta	2.67	3.67	5.33	15.33
Rank	4	3	2	1

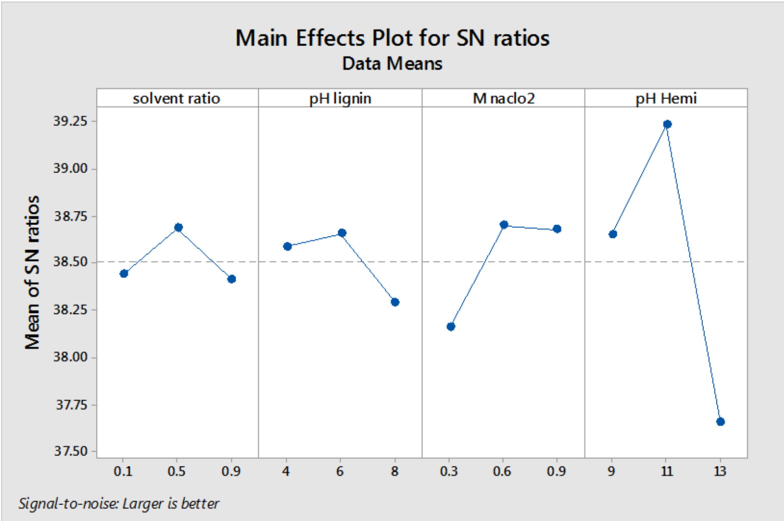

Figure 1: Diagram of signal-to-noise values in the cellulose extraction step

Nanocellulas are classified into three branches according to their size, yield, and method, which in turn depend on cellulosic sources and production conditions[14]. The obtained XRD spectra for the cellulose extracted are shown in Figure 2. According to the results obtained in the extracted cellulose structure, peaks appeared in the 2θ range of 15.657 ° and 22.159 °, respectively, confirming the cellulose structure[15]. Figure 3 presents the results of the FTIR analysis. At 3412cm^{-1}, 3436cm^{-1} and 3438cm^{-1}, a broad absorption peak is observed, indicating the tensile vibrations of the -OH groups in the cellulose structure[16,17]. Also, in the range of 2929 cm^{-1}, 2923 cm^{-1} and 2916 cm^{-1} in the cellulose structure, an absorption peak was observed which could be due to C-H tensile vibrations in the material structure[18]. In the cellulose structure, there is an absorption peak in the range of 1646 cm^{-1}, respectively, which can be due to the absorption of -OH vibrations[19,20]. The absorption peaks were observed in the range of 1237-1437 cm^{-1} and 1057 cm^{-1}, which can be due to the symmetric and asymmetric vibrations of the carboxylic acid spheres[21], H and C-O are in the structure of said materials[22]. The image obtained from the SEM analysis is shown in Fig. 3, which can be confirmed by the results of the nanostructure analysis. As can be seen in the figure, the particle size is nanosized.

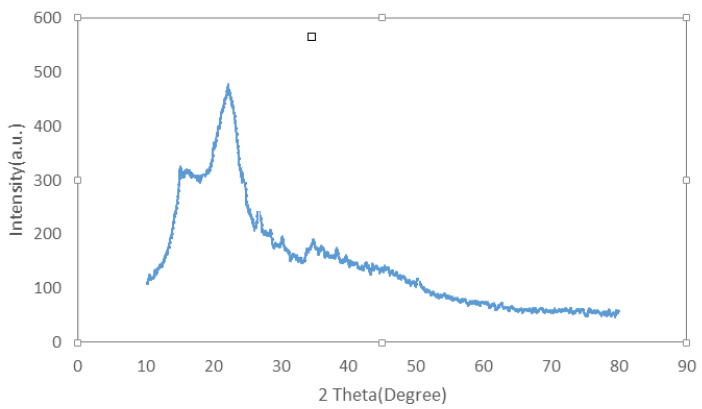

Figure 2

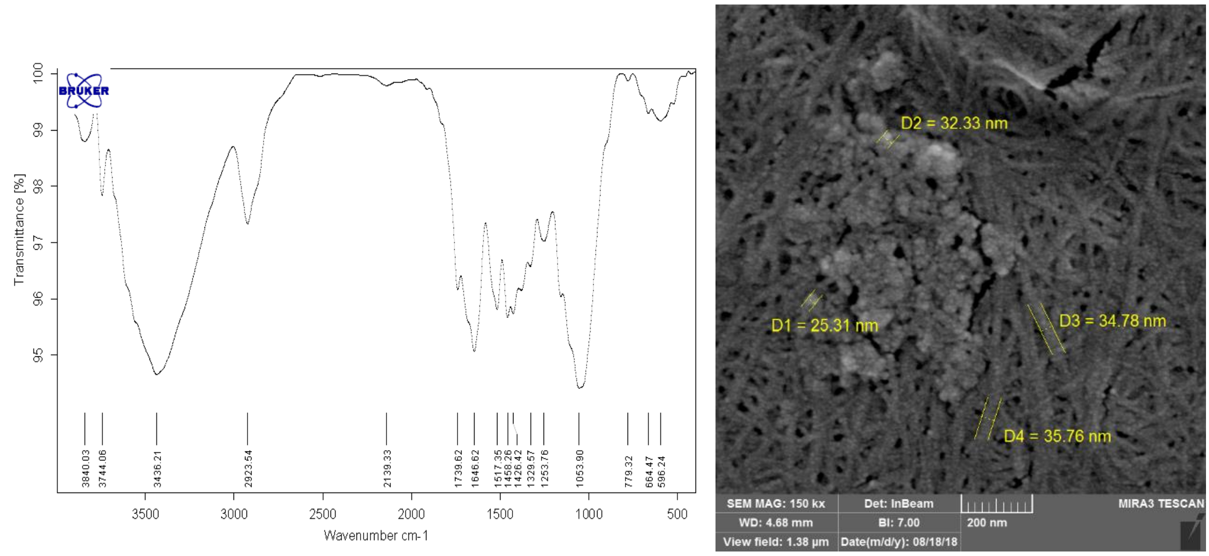

Figure 3

The optimal conditions presented by the Taguchi statistical scheme are presented in Table 4. The predicted extraction rate of Taguchi was 96.66%, which resulted in an extraction rate of 95.68% with two iterations.

Table 4: optimum condition

solvent ratio	pH of Delignification	Sodium chlorite concentration	pH of Hemicellulose removal
83.67	85.33	81.00	85.67

Conclusions

In the present study, the effective parameters on the extraction of cellulose from sugarcane bagasse were optimized by Taguchi statistical analysis of L9 array. The percentage of cellulose extracted under optimum conditions was 95.66%. The cellulosic structures were synthesized using the acidic hydrolysis method extracted by cellulose.

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