Research Article

Extraction Parameters Optimization of Sugarcane Wax from Filter Cake Using Taguchi Method and Grey Relational Analysis

Manunya Okhawilai[1], Pornpen Siridamrong[2] and Supaporn Lekhavat[3,*]

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ABSTRACT

Sugarcane is one of the most important economic crops in Thailand that the country was ranked as the world's second largest exporter. Over the past decade, utilization of by-products such as bagasse, molasses and filter cake had been studied, in order to add value to the product, especially filter cake which contains natural wax. In this research, the sugarcane wax was extracted using the simple methods to provide the high yield and quality of the wax for further using in dental applications. The extraction conditions of sugarcane wax were designed based on Taguchi. Taguchi method with three factors and three levels of solid to liquid ratios (1/15, 1/20 and 1/25 g of filter cake/ml of solvent), extraction time (2, 4 and 6 hr) and extraction temperature (50, 60 and 70°C) were used to obtain high extraction yield and high sugarcane wax quality. Grey relational analysis (GRA) was employed to convert multi-response problem into single response optimization. The results showed that extraction temperature exhibited the highest contribution for the obtained yield. A high solid to liquid ratio and a low temperature of extraction resulted in low quality of sugarcane wax in terms of melting and thermal degradation temperature. The highest extraction efficiency could be achieved at optimum parameter condition using 1 g/15 ml of solid to liquid ratio, 70°C of extraction temperature and 6 hr of extraction time. Such findings from this research indicated that optimization by GRA was an effective tool to provide the highest extraction yield and properties.

Keywords: Sugarcane wax, Filter cake, Multiple response optimization, Taguchi method, Grey relational analysis

[1]Metallurgy and Materials Science Research Institute, Chulalongkorn University, Bangkok 10330, Thailand
[2]2/332 Soi Phahonyothin 40, Sana Nikhom District, Chaktuchak, Bangkok, Thailand
[3]Expert Centre of Innovative Health Food, Thailand Institute of Scientific and Technological Research, 35 Moo 3 Technopolis, Klong Luang, Pathumthani, 12120, Thailand
[*]Corresponding author, email: supaporn_pis@tistr.or.th

1. Introduction

Sugarcane plantation industry is one of the important industries in Thailand directly related to the sugar production industry. Filter cake or press mud is a biowaste that occurs from the process. It was reported that the filter cake was about 300,000 tons/year in Thailand and can be utilized as fuel [1] and fertilization [2]. Besides, the filter cake contains some valuable matters for example oily wax which can be obtained by solvent extraction. Regarding its chemical compositions, sugarcane wax consists of many compounds for example sec-butyl isothiocyanates, alkane heneicosane and fatty acid which are found to beneficial in medicinal and industrial applications. Wax can also be used as food preservative such as an edible coating film for fruits and vegetables [3]. This valuable filter cake thus attracted many researchers to study and create high value-added product from it.

Industrial extraction methods included solvent extraction [4], supercritical CO_2 extraction [5-7] and hydrothermal extraction [8]. Among those, solvent extraction is a simple technique that effectively removes the wax from filter cake. Generally, the conventional extraction of sugarcane wax from filter cake is done commercially using toxic solvents such as benzene, toluene and heptane or strong toxic refining agents such as sulfuric acid, nitric acid and chromic acids [9]. However, the mild, less carcinogenic and less inflammable solvent such as carbon tetrachloride, hexane and other alcohols are alternative choices [10].

For solvent extraction, parameters that affect the extraction yield and properties of the obtained wax are source of material, filter cake to solvent ratio, type of solvent, extraction temperature and extraction time. There were reports that the sugarcane peel and bagasse showed only 0.95% and 1.2% of extraction yield which was much lower than that obtained from filter cake [3, 11]. Extraction time, temperature and type of solvent affected both yield and quality of the sugarcane wax [12]. The solvent consumption is directly related to the cost of extraction process and the amount of solvent waste. A suitable filter cake to solvent ratio is an important parameter to improve the extraction yield and to reduce the waste of solvent.

Optimization is an effective and necessary tool to maximize the extraction efficiency. Taguchi method is one of the optimization techniques which helps reduce the number of experiments, enhance qualities of products and determine design solutions [13-14]. Generally, Taguchi method has been used to determine the optimization of single response. Various extraction systems have been successfully optimized by Taguchi method [14-15]. For optimization of multi response problem, an integration of Taguchi method and Grey relational analysis can convert the multi response results into a relational single grade [16].

Currently, there is no information related to the optimization of extraction conditions of sugarcane wax. The objective of this research is to study and optimize the parameters to extract sugarcane wax from filter cake. Three factors, including filter cake to solvent ratio, extraction temperature and extraction time at each three levels were optimized employing Taguchi method and Gray relational analysis. The properties in terms of extraction yield, melting and degradation temperature and char yield were investigated.

2. Materials and Methods

2.1 Material

Filter cake used in this research was collected from Nakornphet Company, Kamphaeng Phet, Thailand. N-hexane solvent used was analytical grade and purchased from Sigma Aldrich, Singapore. Commercial grade sugarcane wax (graded DEUREX X 51 G) was procured from DEUREX GmbH (Germany).

2.2 Extraction of sugarcane wax

Filter cake of sugarcane is an agricultural waste from the sugar mill factory which still contains some elements that can be added to add value. The influences of dry filter cake to solvent ratio, extraction temperature and time on extraction yield, melting temperature, degradation temperature and char yield are studied as it is known that the harsh extraction conditions may cause a decomposition of the chemical substances resulting in lowered relevant properties. Therefore, the extraction conditions will be optimized to maximize those mention properties of the extracted wax. Filter cake was firstly crushed and grounded into fine powder and filtered using 50 mm diameter mesh. The fine powder was then dried at a temperature of 40°C for 24 hr to remove some of the moisture which might cause fermentation. The weighed filter cake and solvent were put into 1000 ml round bottle flask equipped with condenser. The distillation equipment was then heated using heating mantle. The hot extraction was filtered using a funnel and then evaporated to dryness. The obtained wax was weighed and kept at -20°C. The factors and factor levels for extraction are shown in Table 1. The extraction conditions were designed using Taguchi method. The conditions for extraction of sugarcane i.e. solid to liquid ratio (factor A = 1/15, 1/20 and 1/25 g ml^{-1}), extraction temperature (factor B = 50, 60 and 70°C), and extraction time (factor C = 2, 4 and 6 hr) are given in Table 2. The obtained wax was then characterized without purification. Experiments were done in triplicate.

Table 1 Factors and factor levels.

Levels	Factors		
	Solid to liquid ratio, factor A (g. dry filter cake/ml of solvent)	**Extraction temperature, factor B (°C)**	**Extraction time, factor C (hr)**
1	1/15	50	2
2	1/20	60	4
3	1/25	70	6

2.3 Characterization

The %yield of extracted sugarcane wax via different conditions was calculated according to the Eq (1)

$$\%\text{yield of extraction} = \frac{\text{dry weight of extracted sugarcane wax}}{\text{dry weight of filter cake}} \times 100 \qquad (1)$$

The thermal behaviors of the obtained wax samples were investigated using differential scanning calorimeter (DSC) model DSC1, METTLER TOLEDO, Thailand. The changes in heat flow was performed at a temperature range of 30-105°C at a heating rate of 10°C/min under nitrogen atmosphere. Sample was first heated from 30 to 105°C at a heating rate of 10°C/min, held at 105°C for 10 min, and cooled down to 30°C at a cooling rate of 10°C/min. DSC curves of the last heating cycle were used to determine the melting point range.

The thermal stabilities of the wax were observed using Thermogravimetric analyzer (TGA) model TGA1, METTLER TOLEDO, Thailand. The initial weight of the sample to be tested was about 10 mg. It was heated from a room temperature to 600°C at a heating rate of 20°C/min under nitrogen atmosphere. The degradation temperature at 10% weight loss and solid residue determined at 600°C were recorded for each sample.

Table 2 Design of experiment of sugarcane wax extraction from Taguchi method.

Sample	Solid to liquid ratio (g of dry filter cake/ml of solvent)	Extraction temperature (°C)	Extraction time (hr)
1	1/15	50	2
2	1/15	60	4
3	1/15	70	6
4	1/20	50	4
5	1/20	60	6
6	1/20	70	2
7	1/25	50	6
8	1/25	60	2
9	1/25	70	4

Taguchi method containing three factors and three levels was used to optimize the conditions of UAE process in terms of single response and multiple responses. The conditions and their variation levels are shown in Table 2. The signal to noise ratio (S/N) was used to evaluate the effect of each parameter level for single response optimization with the help of ANOVA.

The S/N ratios were classified into three classes: (1) nominal-the-better, (2) smaller-the-better, and (3) larger-the-better which were applied for optimization [17]. In this study, all responses including extraction yield, melting temperature, degradation temperature and char yield were maximized regarding to the "larger is better" and S/N ratio was analyzed based on Eq. (2).

$$(\mathrm{S/N}) = -10 \log \left[1/R \sum_{f=1}^{R} 1/y_i^2 \right] \tag{2}$$

where R is the number of all data points and yi is the value of i^{th} data point.

The Grey relational analysis was used to convert multiple responses optimization into the single response optimization by S/N ratios calculation. The obtained results from Taguchi method can then be calculated to determine the highest overall grey relational analysis which represents the optimal parametric combination. Before grey relational analysis, data preprocessing is normally required, that is a process of transferring the original sequence to a comparable sequence that is normalized within the range of zero to one [18]. The reference sequence and comparable sequence can be denoted by $x_0(k)$ and $x_i(k)$ for i = 1, 2, ..., m; k = 1, 2, ..., n, respectively, where m is the total number of experiments to be considered, and n is the total number of observation data. The appropriate equation for the normalization also depends on the type of the quality characteristic. In this work, the-larger-the-better quality characteristic was applied for the normalization of all responses that as expressed in Eq. (3).

$$x_i(k) = \frac{y_i(k) - \min y_i(k)}{\max y_i(k) - \min y_i(k)} \quad (3)$$

where x_i(k) is the value after grey relational generation,

min y_i(k) is the smallest value of y_i(k) for k^{th} response, and

max y_i(k) is the largest value of y_i(k) for k^{th} response.

Grey relational coefficient can be calculated using Eq. (4).

$$\gamma\left(x_0^*(k), x_i^*(k)\right) = \frac{\Delta_{\min} + \zeta\Delta_{\max}}{\Delta_{0i}(k) + \zeta\Delta_{\max}} \quad (4)$$

$0 < \gamma\left(x_0^*(k), x_j^*(k)\right) \leq 1$

where $\Delta_{0i}(k) = \left|x_0^*(k) - x_i^*(k)\right|$,

$$\Delta_{\max} = \underset{\forall j \in i}{\max}.\ \underset{\forall k}{\max}.\ \left|x_0^*(k) - x_j^*(k)\right|$$

$$\Delta_{\min} = \underset{\forall j \in i}{\min}.\ \underset{\forall k}{\min}.\ \left|x_0^*(k) - x_j^*(k)\right|,$$

ζ is the distinguishing coefficient, $\zeta \in [0,1]$.

If all process parameters have equal weighting, ζ is set to be 0.5. The grey relational grade is the average of all grey relational coefficients, which can be determined using Eq. (5).

$$\gamma\left(x_0^*, x_i^*\right) = \frac{1}{n}\sum_{k=1}^{n} \beta_k \gamma\left(x_0^*(k), x_i^*(k)\right) \quad (5)$$

Finally, the optimal condition of sugarcane wax extraction can be the level corresponding to the highest value of average gray relational grade of each factor.

3. Results and discussion

3.1 Extraction yield of the sugarcane wax

The color of wax of all samples was brownish green. The yield values and S/N ratio of sugarcane wax extracted regarding to Taguchi's experimental design are shown in Figure 1.

As seen in Figure 1, the extraction yield of sugarcane wax tended to decrease with increasing filter cake/solvent ratio. It is also noticed from Figure 1 that the yield value increased at higher extraction temperature. The greater in yield value with extraction temperature from

50 to 70°C accounted to about 20%. It was because the higher extraction temperature of 70°C is likely closed to the melting temperature of the sugarcane wax resulting in the dissolution of the wax into solvent [10]. In contrast, a longer extraction time resulted in a lower yield value. As an extraction time increased from 2 to 4 hr, the yield increased from 5.46 to 5.69% then decreased to 5.28% at extraction time of 6 hr. The simple solvent extraction used in this research provided the extraction yield higher than using reflux with carbon tetrachloride having the value in the range of 3.5 to 4.10% [10]. The yield value of sugarcane wax extracted using n-hexane was in the same range of that using Soxhlet extraction with toluene having the values of 5.40 to 5.60% [4]. In comparison to other extraction methods, the yield obtained from this research was much higher than that of sugarcane wax extracted using supercritical CO_2 fluid [5]. It can be concluded that an extraction technique is simple, easy manipulation which provide relatively high yield compared to other extraction methods. The best extraction condition was at solid/liquid ratio of 1/15, extraction temperature of 70°C and extraction time of 4 hr.

The S/N ratio showed the similar trend to the yield values of each parameter i.e. the ratio also decreased with increasing solid/liquid ratio where the S/N ration increased with increasing extraction and the yield value increased initially then drop with time in the same manner to the yield obtained with extraction time.

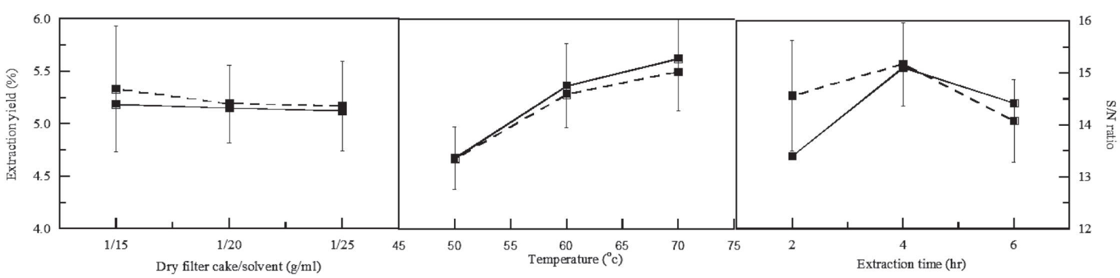

Figure 1 Effect of extraction condition on yield of sugarcane wax: yield (solid line) and S/N ratio (dash line).

With multi-linear regression analysis, the extraction yield was formulated as a function of extraction condition as seen in Eq. (6).

$$\text{Extraction yield (\%)} = 1.457 + 6.24X_1 + 0.0478X_2 + 0.144X_3 \qquad R^2 = 0.77 \qquad (6)$$

Where X_1, X_2 and X_3 represent dry filter cake/solvent (g ml^{-1}), temperature (°C) and extraction time (hr), respectively. It appeared that an extraction temperature was the main factor influencing their yields at relatively high %contribution as shown in Table 3.

Table 3 Result of ANOVA analysis.

Properties	Factor		
	Dry filter cake/solvent(g ml^{-1})	Temperature ($^{\circ}C$)	Extraction time (hr)
Extraction yield			
Degree of freedom	3	3	3
SS*	0.11	1.53	0.50
v**	0.05	0.76	2.50
Contribution (%)	5.06	71.53	23.41
Melting temperature			
Degree of freedom	3	3	3
SS*	15.65	16.76	9.73
v**	7.82	8.38	4.86
Contribution (%)	37.14	39.77	23.09
Degradation temperature			
Degree of freedom	3	3	3
SS*	112.47	29.05	132.188
v**	56.09	10.61	48.30
Contribution (%)	41.09	10.61	48.30
Char yield			
Degree of freedom	3	3	3
SS*	4.66	0.65	0.32
v**	2.33	0.32	0.16
Contribution (%)	82.70	11.57	5.73

3.2 Melting temperature of the sugarcane wax

The melting temperature is one of the most important properties of wax relating to the usage conditions upon heating. The higher melting temperature of the wax implies the easy to carry out during fabrication. The effects of dry filter cake to solvent ratio, extraction temperature and time on extraction yield on melting temperature of the extracted wax were investigated. The melting temperature and S/N ratio of the extracted sugarcane wax regarding to Taguchi's experimental design is shown in Figure 2.

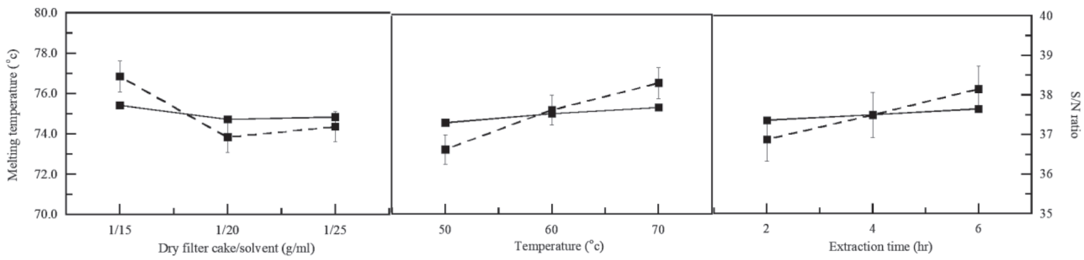

Figure 2 Effect of extraction condition on melting temperature of sugarcane wax: melting temperature (solid line) and S/N ratio (dash line).

As seen in Figure 2, the melting temperature of the extracted wax tended to decrease with increasing dry filter cake to solvent ratio. The S/N ratio also showed the similar trend to the melting temperature. The effect of extraction temperature on melting temperature of the wax was considered. The values slightly increased with increasing extraction temperature whereas, the S/N ratio sharply increased with increasing extraction temperature. The melting temperature of sugarcane wax was observed to increasing with extraction time. The S/N ratio was also observed to have the similar trend. The best of each condition to provide the maximum melting temperature were at 1/15 filter cake to solvent ratio, 70°C temperature and 6 hr. The sugarcane wax extracted from this research showed the melting temperature to be slightly higher than that extracted using reflux technique with carbon tetrachloride having the value of about 72 to 75°C [10]. It may be due to the use of mild and safety hexane solvent which did not deteriorate the property of the extracted sugarcane wax. Interestingly, the sugarcane wax extracted from filter cake showed higher melting temperature than that of extracted from sugarcane peel which was reported to be about 62.12°C [3]. The result indicated the higher melting temperature of the wax in the inner layer of sugarcane than that of the outer layer of peel. With multi-linear regression analysis, the melting temperature was formulated as a function of extraction condition as seen in Eq. (7).

$$\text{Melting temperature } (^{\circ}\text{C}) = 57.128+102.78X_1+0.166X_2+0.637X_3 \quad R^2 = 0.85 \quad (7)$$

Where X_1, X_2 and X_3 represent dry filter cake/solvent (g ml^{-1}), temperature (°C) and extraction time (hr), respectively. Table 3 indicated that all three parameters affected their melting temperature in the equal level. However, the filter cake to solvent ratio showed the highest %contribution.

3.3 Thermal stabilities of the sugarcane wax

Degradation temperature of the sugarcane wax at 10% weight loss and char yield at 600°C were carried out in this experiment. Those mentioned values implied the thermal stabilities of the extracted wax under an elevated temperature. The degradation temperatures and S/N ratios obtained from each extraction condition was displayed as seen in Figure 3. As filter cake to solvent ratio increased, the degradation temperature decreased. The S/N ratio also dropped with increasing filter cake to solvent ratio. The higher thermal properties including melting and degradation temperatures were noticed with lower filter cake to solvent ratio. Whereas, the degradation temperature of the wax initially increased with increasing extraction temperature then slightly dropped as the temperature increased. The S/N ratio also exhibited the same trend to the degradation temperature. In contrast, the degradation temperature of the sugarcane wax was obviously increased with extraction time. Similarly, the S/N ratio showed the same trend to degradation temperature i.e. the S/N ratio increased with time. Consequently, the most suitable extraction condition to provide the maximum degradation temperature of each parameters were at 1/15 filter cake to solvent ratio, 60°C extraction temperature and 6 hr extraction time.

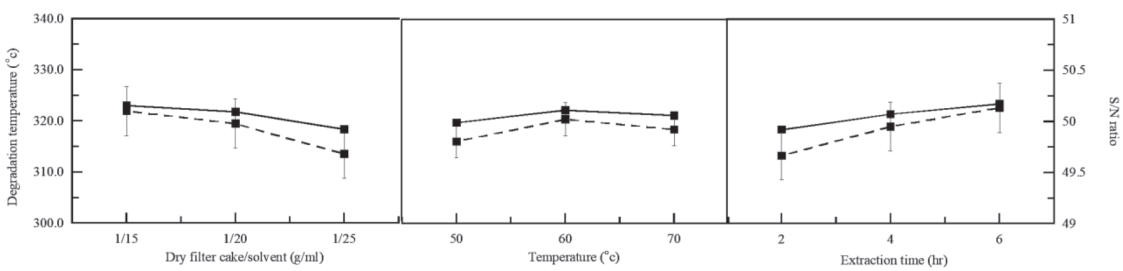

Figure 3 Effect of extraction condition on degradation temperature of sugarcane wax: degradation temperature (solid line) and S/N ratio (dash line).

With multi-linear regression analysis, the degradation temperature was formulated as a function of extraction condition as seen in Eq. (8).

$$\text{Degradation temperature } (^\circ\text{C}) = 286.34+298.01X_1+0.118X_2+286.34X_3 \qquad R^2 = 0.87 \qquad (8)$$

Where X_1, X_2 and X_3 represent dry filter cake/solvent (g ml^{-1}), temperature (°C) and extraction time (hr), respectively. The extraction time showed the most contribution to their thermal degradation among the others parameter as shown in Table 3.

The char yields and the S/N ratios of the sugarcane wax are illustrated in Figure 4. The char yield dropped as filter cake to solvent ratio increased. The result implied that this extraction method was environmentally friendly and low cost of production to use lower amount of solvent used for extraction. The S/N ratios also showed the same trend to as char yield value. The extraction temperature of the sugarcane resulted in an increase of char yield. As extraction time increased, the char yield slightly increased then decreased. The S/N ratios also showed the same trend to as char yield value for all extraction parameters. The best extraction conditions for each parameter to provide the maximum char yield were at solid to solvent ratio of 1/20, extraction temperature at 70°C and extraction time of 4 hr.

With multi-linear regression analysis, the degradation temperature was formulated as a function of extraction condition as seen in Eq. (9).

$$\text{Char yield (\%)} = 2.739+14.3367X_1+0.0228X_2-0.992X_3 \qquad R^2 = 0.87 \qquad (9)$$

Where X_1, X_2 and X_3 represent dry filter cake/solvent (g ml^{-1}), temperature (°C) and extraction time (hr), respectively. From Table 3, the char yield of the sugarcane wax strongly depended on solid to solvent ratio.

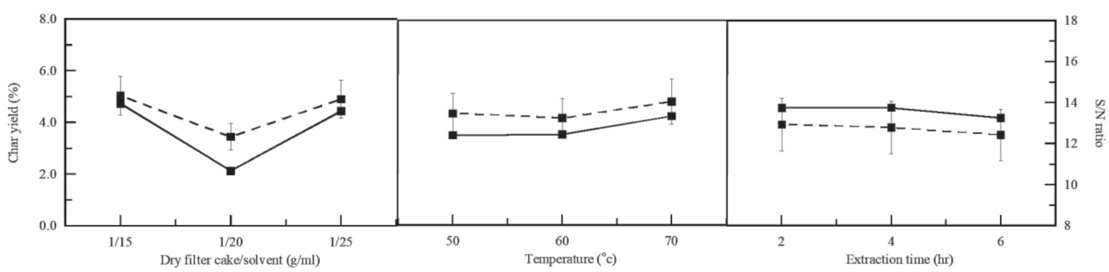

Figure 4 Effect of extraction condition on char yield of sugarcane wax: char yield (solid line) and S/N ratio (dash line).

3.4 Optimum extraction condition of sugarcane wax using Grey relational analysis

The overall properties including extraction yield, melting temperature, degradation temperature at 10% weight loss and char yield of sugarcane wax were important for an ideal design with the suitable extraction condition of the wax. Therefore, it is necessary to optimize the extraction conditions of sugarcane wax while maintaining such relevant properties at acceptable values. For this purpose, Grey relational analysis was applied to convert a multi response objective problem into a single result. The results of Grey analysis are summarized in Table 4 and Table 5. The larger Grey relational grade implied that the comparability sequence has the highest degree of similarity with the reference sequence. Therefore, the highest value of Grey relational grade indicates the optimum of each parameter. From the table, experiment number 3 showed the highest Grey relational grade values consisting of A_1, B_3 and C_3. Therefore, the optimal extraction condition of sugarcane wax was obtained at solid to solvent ratio of 1/15, temperature at 70°C and extraction time at 6 hr.

Table 4 The calculated Grey relational coefficient and Grey relational grade for nine comparability sequence.

Experimental run	Grey relational coefficient				Grey relational grade
	Extraction yield	Melting temperature	Degradation temperature	Char yield	
1	0.33	0.42	0.35	0.35	0.36
2	1	0.62	0.99	0.58	0.79
3	0.99	1	1	0.52	0.88
4	0.60	0.33	0.48	1	0.60
5	0.52	0.50	0.70	0.61	0.58
6	0.55	0.42	0.43	0.84	0.56
7	0.45	0.41	0.46	0.66	0.50
8	0.51	0.37	0.33	0.59	0.45
9	0.77	0.54	0.34	0.33	0.49

Table 5 The values of Grey relational analysis.

Levels	Dry filter cake/solvent ratio: A	Temperature: B	Extraction time: C
1	0.6808	0.4879	0.4595
2	0.5838	0.6119	0.6334
3	0.4816	0.6464	0.6533

The confirmation test was performed to determine the accuracy of the optimal combination. The predicted optimal value of the sugarcane wax can be calculated as shown in Eq. (10).

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^{q} (\gamma_i - \gamma_m) \tag{10}$$

Where γ_m is the total mean of Grey relational grade, γ_i is the mean of Grey relational grade at optimal level and q is the number of machining parameters that significantly affected multiple performance characteristics. The actual value was in good agreement with the predicted value, as displayed in Table 6.

Table 6 Results of confirmation experiment.

	Predicted value	**Experiment value**
Grey relational grade	0.8163	0.8790

4. Conclusions

In this research, the valuable sugarcane wax was extracted from filter cake which was biowaste from sugarcane juice production using n-hexane solvent extraction. Effects of extraction conditions including filter cake to solvent ratio, extraction temperature and extraction time on yield, melting and degradation temperatures and char content of the sugarcane wax were investigated. Single response was used for optimization. It was found that extraction temperature, filter cake to solvent ratio strongly affected the obtained yield and char yield, respectively, where melting and degradation temperatures slightly affected the extraction temperature and time, respectively. Grey relational analysis (GRA) which was used to convert multi response to single response showed that the optimal extraction condition of sugarcane wax was at solid to solvent ratio of 1/15, temperature of 70°C and extraction time of 6 hr. In summary, the extraction condition from this research could be further used for sugarcane wax extraction from filter cake to meet those properties requirement for wide range of applications.

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