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Research article

Optimization of iodine number of carbon black obtained from waste tire pyrolysis plant via response surface methodology

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ABSTRACT

Recovered carbon black (RCB) obtained from a tire pyrolysis plant was subjected to chemical and thermal treatments for application as a filler in rubber compounds. Carbon black was chemically treated with nitric acid by varying the temperature, time, and chemical-to-carbon black ratio. The iodine number was optimized using response surface methodology (RSM) and the Design Expert software. To increase the iodine number, the Box–Behnken design was utilized to optimize three parameters: temperature (30–50 °C), time (6–24 h), and ratio of carbon black to chemical (0.25–1.0 g/mL). Under optimal conditions, the surface area increased, and RCB was upgraded to commercial carbon black N330. RSM analysis indicted that the iodine number was maximized (117.34 mg/g) after treatment at 46.74 °C for 23.24 h using a carbon black/chemical ratio of 0.76 g/mL. The simulated data were experimentally validated by analyzing RCB_ EQ, which yielded an iodine number of 119.12 mg/g. The content of most heavy metals in RCB decreased by more than 90%, whereas the sulfur and chlorine content decreased by 43.27% and 53.96%, respectively. Based on thermogravimetric analysis, the RCB_13 carbon black additive was eliminated at temperatures of 620–800 °C.

1. Introduction

Owing to the increasing accumulation of waste tire and demand for industrial fuel oil, the use of waste tire pyrolysis in Thailand has increased. However, the high temperatures and lengthy residence durations necessary for this method restrict the synthesis of polyaromatic hydrocarbons, while aliphatic hydrocarbons are generated via Diels-Alder reactions [1]. Additional challenges, including product market limitations, legislative barriers, and the high proportion of impurities in the resulting char, currently limit the use of the char in high-value recycled materials [2, 3]. Because the composition of the tire feedstock has a wide range of quality-affecting variables, such as the fixed carbon, ash, and volatile matter content, the quality of recovered carbon black (RCB) is negatively impacted by the high ash content of the tires, resulting in a skewed distribution of these variables [4].

Carbon black is a paracrystalline carbon form which results when the combustion of plants matter, heavy oil, fuel oil obtained from petroleum is incomplete. It can also be produced through the process of fluid catalytic tar or ethylene cracking. It provides a high ratio of surface area to volume, although this does not exceed that of activated carbon. However, the ratio is much higher than that of soot, while carbon black also differs from soot in having notably lower polycyclic aromatic hydrocarbon (PAH) content, to the extent that it is non-bioavailable. Accordingly, carbon black serves as an effective model compound in the process of fuel oxidation, and also finds use as reinforcement filler for rubber products such as tires. It can be employed as a colorant, as an additive to prevent wear in plastic products, or as a pigment in paints or inks [5]. The naming system ASTM D1765-16 is used to classify rubber-grade carbon black. The initial character in this system of nomenclature shows how carbon black affects the curing rate of different rubber compounds. The average surface area of carbon black is indicated by the second character. The assignments of the last two characters are unconfirmed [6]. The most common grade of carbon black rubber is N330, which has an iodine content of 82 g/kg.

RCB is a solid by-product of waste tire pyrolysis. RCB differs considerably from commercial carbon black (CCB) in that it has a significantly different structure and contains undesirable components, include carbonaceous occurrences and inorganic compounds [7]. As the

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Table 1. Summary of RSM design.

Factor	Name	Unit	Level		
			Low	Middle	High
A	Temperature	°C	30	40	50
B	Ratio	g/mL	0.25	0.5	1.0
C	Time of reaction	h	6	12	24
Response	**Name**	**Unit**			
Y	Iodine number	mg/g			

fraction of carbon black in RCB increases, certain properties match those of pure CCB, compensating for the low reinforcement properties of RCB [8, 9]. It was demonstrated that the reinforcing effect and cross-linking density of RCB-filled vulcanized materials were marginally inferior to those of conventional carbon black (N550), where RCB was effective as a reinforcement in natural rubber composites. Based on scanning electron microscopy (SEM) analysis, the particle shape of RCB differs from that of CCB. RCB has a smaller surface area than CCB N330 carbon black [10].

Acidic and basic solutions are used to dissolve inorganic impurities in the demineralization of RCB. Acid demineralization is more effective in dissolving inorganic impurities than base demineralization [11, 12]. Acid solutions, such as HNO_3, HCl, H_2SO_4, and HF, have been used for demineralization. Nitric acid dissolves Ni and V more efficiently than sodium hydroxide [13]. Hydrochloric acid is superior to sulfuric acid for dissolving zinc [12], and the agglomerate size of CCB is larger than that of RCB after acid treatment owing to carbonaceous deposits formed during pyrolysis [14].

When experiments are carried out, every variable with one exception will be maintained at a constant level, while the remaining variable will undergo systematic changes in order to obtain the resulting data. This approach is known as the 'one variable at a time' (OVAT) method. However, one drawback to this approach is that the effects upon the response of multiple variable changes in combination cannot be quantitatively assessed [15]. OVAT techniques for optimizing the operation of diesel engines are time consuming and expensive [16]. Process conditions are optimized using Response Surface Methodology (RSM), a multivariate statistical approach ideal for modeling complicated processes [17]. Owing to the multivariate statistical nature of RSM, this method exhibits a high performance when the experimental response is affected by numerous variables. Numerous researchers have used RSM to optimize biodiesel production processes and study the effect of each parameter on maximizing biodiesel production [18, 19, 20]. Design Expert is the most widely used software for determining the optimal point. The design-of-experiment-specific (DOE) software package provided by Stat-Ease Incorporation is a statistical application. Comparative assessments, screening, characterisation, optimization, resilient parameter design, mixture design, and integrated design are all made possible by Design-Expert [21]. Design-Expert offers screening matrices for up to 50 different factors. To evaluate if these variables are statistically significant, analysis of variance (ANOVA) is utilized. The effect of each component on the desired outcomes may be determined with the use of tools for displaying and identifying anomalies in the data [22]. Carbon black derived from the pyrolysis of waste tire, as a solid waste, can be activated to increase the iodine number to that of N330 carbon black for use as a filler in rubber compounds. Therefore, the objective of this study is to enhance the quality of RCB by removing impurities through acid treatment and increasing the surface area by maximizing the iodine number. The optimal conditions for this process are resolved using RSM. This novel technique is used to predict and optimize the chemical treatment parameters for maximizing the iodine number of RCB by comparison with that of CCB.

2. Materials and method

2.1. Materials

Carbon black resulting from waste tire pyrolysis was produced by Pyro Energies Company Limited in Thailand using a rotary kiln reactor with a final temperature of 450 °C; the iodine number was 34.4 mg/g. Nitric acid (65%, EMSURE) and HF (48%, KEMAUS) were used for the chemical treatment of the RCB; the chemicals were analytical grade reagents.

2.2. Preparation of activated carbons and their subsequent characterization

The powder obtained after tire pyrolysis was sieved through a no. 425 μm mesh, dried in an oven at 150 °C for 1 h, and placed in a glass desiccator. Carbon black was soaked in 48% HF overnight, washed with distilled water until a constant pH was achieved, and then dried in an oven at 110 °C for 24 h. Demineralization was performed with 50% nitric acid under reflux; the conditions were varied as summarized in Table 1. Distilled water was then used to rinse the RCB and wash off any remaining acid, until the filtrate reached a constant pH value. It was then oven-dried for 24 h at a temperature of 110 °C before being placed inside a desiccator. The treated carbon samples were heated at 850 °C in a muffle furnace for 30 min, cooled to room temperature, and placed in a bag with a zipper.

2.3. Characteristics of carbon black

With a heating rate of 10 °C/min and a N_2 flow rate of 40 ml/min in the 30–900 °C temperature range depicted in Figure 1, thermogravimetric analysis (TGA) was carried out using a TGA/DSC3+ (Mettler Toledo) equipment. Fourier-transform infrared (FTIR) spectroscopy data were acquired using a Thermo Scientific (USA) model Nicolet iS50 FTIR

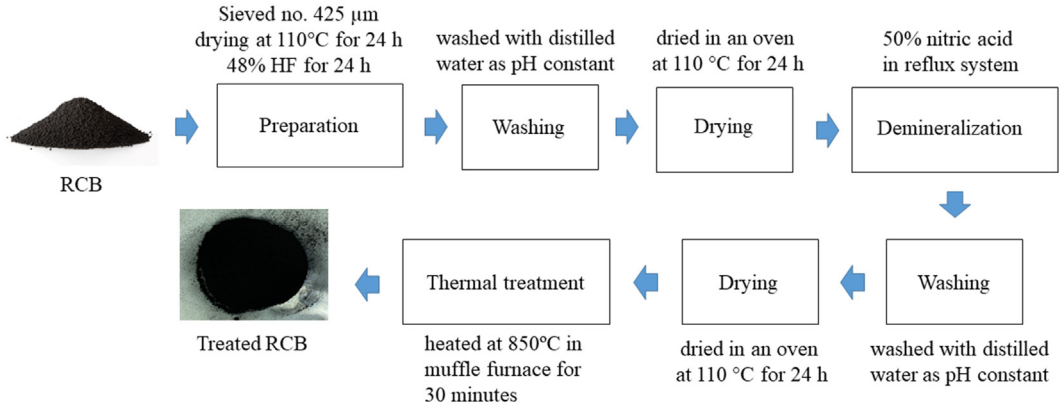

Figure 1. Flowchart of the chemical treatment of RCB.

Table 2. Experimental design results.

Std	Run	A: Temperature °C	B: Ratio g/mL	C: Time h	%Yield	Actual mg/g	Predicted mg/g
11	1	40	0.25	24	78	111.15	111.25
13	2	40	0.5	12	74	108.43	109.77
4	3	50	1	12	88	108.6	108.32
17	4	40	0.5	12	76	110.10	109.77
14	5	40	0.5	12	75	110.88	109.77
9	6	40	0.25	6	75	112.26	111.54
1	7	30	0.25	12	84	101.37	101.62
3	8	30	1	12	60	94.80	94.46
15	9	40	0.5	12	72	110.65	109.77
12	10	40	1	24	71	110.30	110.66
7	11	30	0.5	24	84	103.10	102.89
2	12	50	0.25	12	55	99.50	99.86
8	13	50	0.5	24	55	117.34	117.08
16	14	40	0.5	12	72	108.80	109.77
5	15	30	0.5	6	82	111.59	111.87
10	16	40	1	6	69	112.56	112.82
6	17	50	0.5	6	76	109.76	109.94

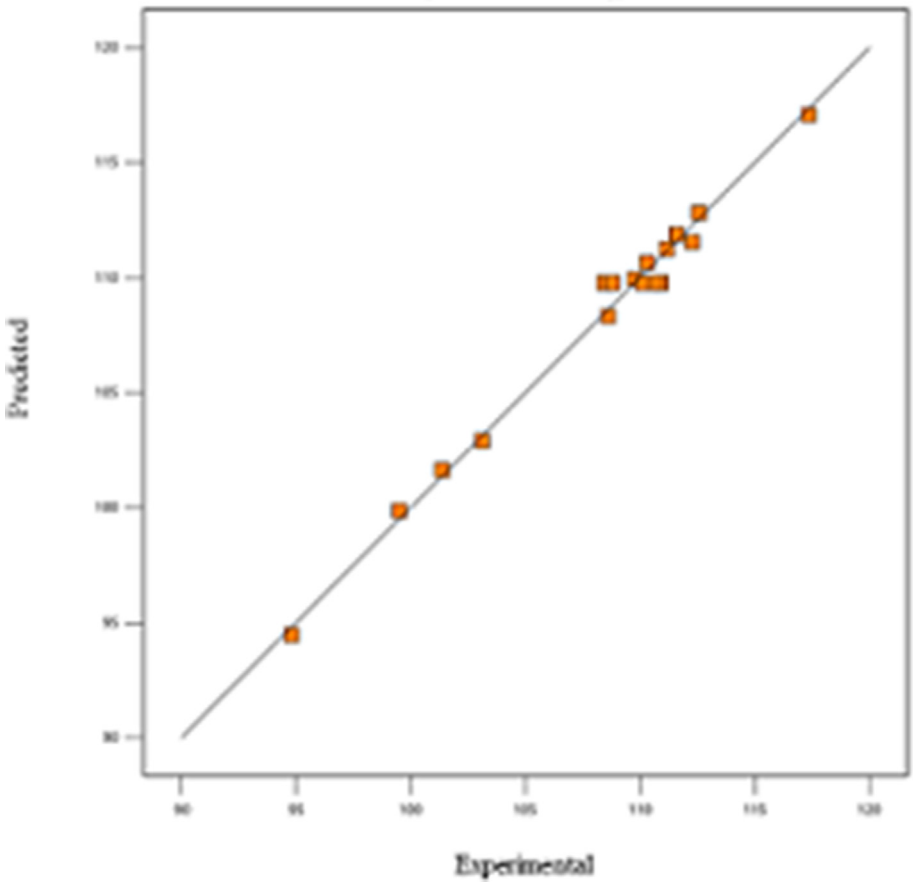

Figure 2. Predicted versus experimental iodine numbers of carbon black.

spectrometer. Elemental analysis was conducted using X-ray fluorescence (XRF) by employing a XOS Petra MAX system. The iodine number was measured according to ASTM 1510-16. The iodine adsorption number, which relies on the surface porosity and may be used to describe the carbon black's surface area. As specified in ASTM D 1510-16, a known amount of iodine was added to the carbon black. The mixture was shaken and centrifuged. Excess iodine was then titrated with a standard sodium thiosulfate solution to determine the adsorbed iodine.

2.4. Experimental design

A three-level, three-factor Box–Behnken design was utilized herein, necessitating 17 experiments. However, these designs are not based on factorial designs, either full or fractional. The design points are located in the middle of the dimension k-1 sub-areas. For example, in the case of the three variables, the points were situated in the middle of the experimental domain edges. The responses of the studied variables were

Table 3. ANOVA for the response surface quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	% Contribution	
Model	484.99	9	53.89	61.13	<0.0001		significant
A-Temperature	137.18	1	137.18	155.61	<0.0001	24.14	
B-Ratio	0.2195	1	0.2195	0.2490	0.6331	0.04	
C-Time	2.86	1	2.86	3.24	0.1147	0.50	
AB	64.19	1	64.19	72.81	<0.0001	11.30	
AC	68.40	1	68.40	77.59	<0.0001	12.04	
BC	0.9672	1	0.9672	1.10	0.3297	0.17	
A^2	102.38	1	102.38	116.13	<0.0001	18.02	
B^2	59.72	1	59.72	67.75	<0.0001	10.51	
C^2	132.26	1	132.26	150.02	<0.0001	23.28	
Residual	6.17	7	0.8816				
Lack of Fit	1.32	3	0.4397	0.3625	0.7846		not significant
Pure Error	4.85	4	1.21				
Cor Total	491.16	16					

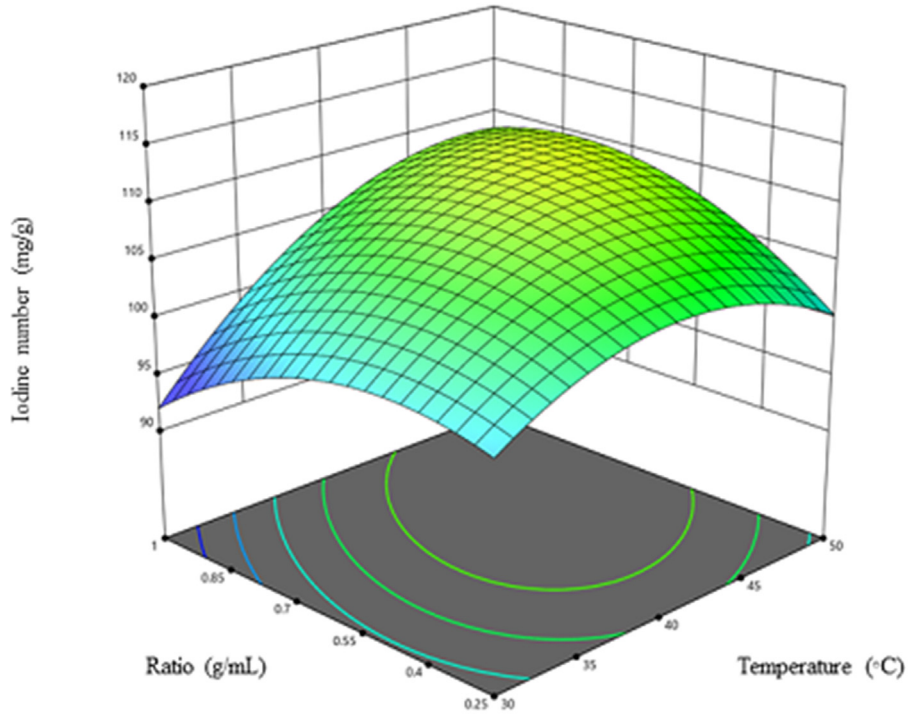

Figure 3. Effect of the interaction between temperature (A) and ration (B) on iodine number.

analyzed according to the response surface. RSM typically uses a second-order model shown in Eq. (1) [23]:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ij} X_i X_j + \sum \beta_{ii} X_i^2 \quad (1)$$

where Y is the iodine number of carbon black for each combination of factors, namely, the response value; the actual value of each variable X is considered, and $X_i X_j$ is the interaction between the independent variables. X_i^2 is the quadratic term of each variable; β_o, β_i, β_{ii}, and β_{ij} are the regression coefficients of the model and are constants. The β coefficients were computed using the least squares technique. RSM can then be applied to determine the values for the parameters which can be adjusted to optimize the response, or to identify those X values which can obtain a particular process or product, in accordance with various requirements or specifications, representing the error associated with the response of Y.

2.5. Analysis of the data

Experimental data underwent analysis with Design Expert 13 software (Stat-Ease Inc., Minneapolis, USA). In order to determine the optimal conditions, three key steps were performed, opening with analysis of variance (ANOVA), before conducting regression analysis, and the plotting of the response surface for use with a typical response for no transform data ($\lambda = 1$)

2.6. Optimization and prediction equation

For each model, a set comprising the optimized reaction conditions was created on the basis of the objective function termed 'desirability' (D). This process made use of the Design Expert software which supports numerical optimization. Analysis of the desirability function analysis

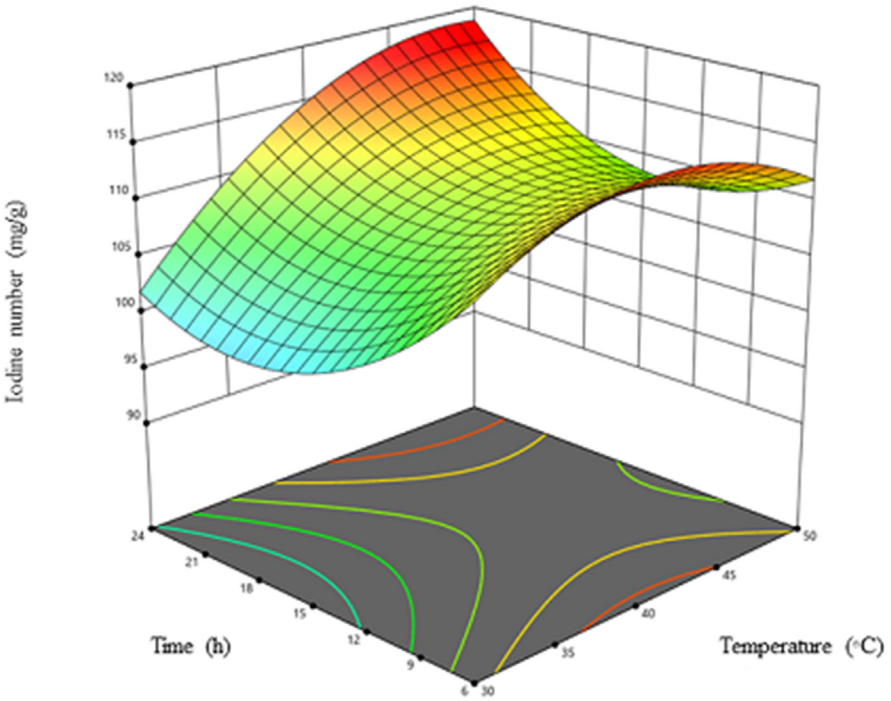

Figure 4. Effect of the interaction between temperature (A) and time (C) on iodine number.

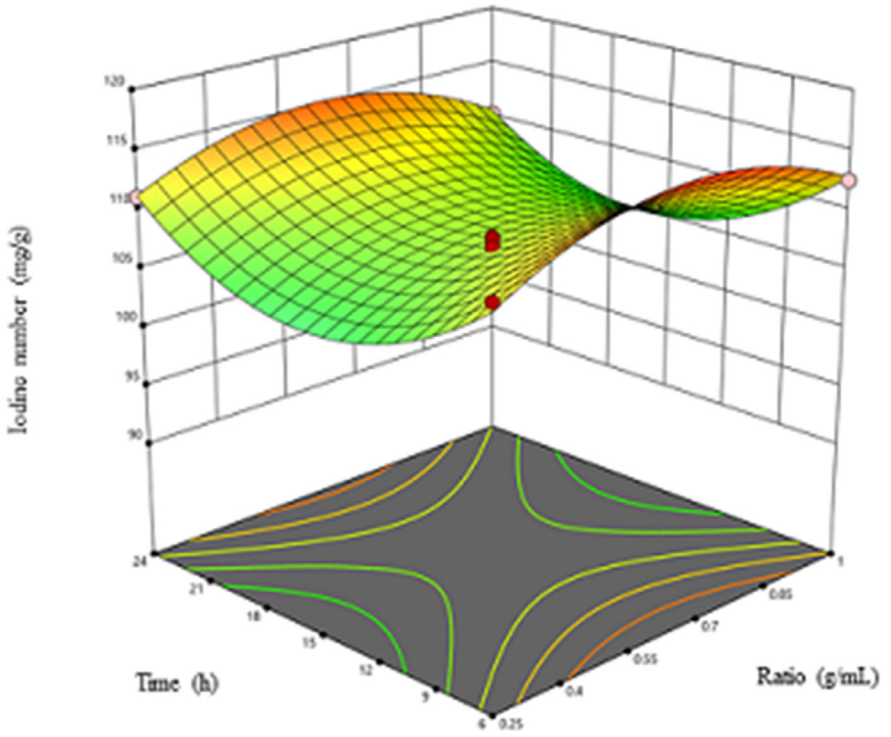

Figure 5. Effect of the interaction between ratio (B) and time (C) on iodine number.

serves as an estimation approach by which the response values are transformed into the interval $0 < D < 1$, for which elevated D values show preferable response values [23]. Accordingly, validation of the RSM model was achieved through the close agreement obtained between the predicted iodine number response values and those reported in the experiment.

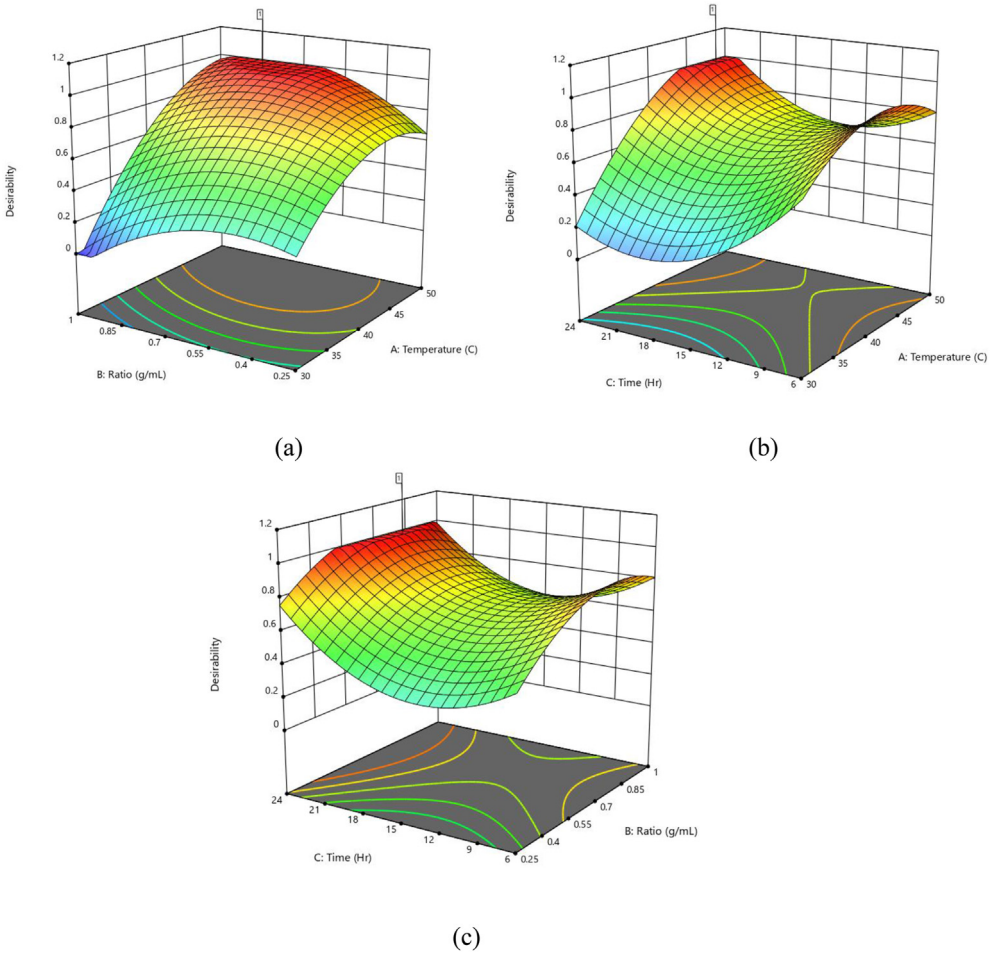

Figure 6. Response surface plot estimated using the desirability function approach.

3. Results and discussion

3.1. Optimization of iodine number using the response surface method

3.1.1. Experimental design and results

The Design Expert 13 software was used for the statistical evaluation of the experimental data for carbon black derived from waste tire pyrolysis. The demineralization of carbon black obtained from the waste tire pyrolysis process has been widely studied. A HCl/char ratio of 5–30 ml/g, demineralization time of 6–24 h, acid concentration of 1–3 M, and thermal treatment temperature of 900–950 °C were investigated as the conditions for preparing activated carbon [24]. Treatment with sulfuric acid was studied, using an acid/carbon black ratio of 3–12 ml/g, acid concentration of 0.10–9.80 N, heating temperature of 20–60 °C, reaction time of 0–60 min, and sulfuric acid recycling number of 1–3 times [25]. The consumption of reagents during the demineralization process is correlated with the reagent-to-carbon black ratio, which is a significant variable [7]. High temperatures, however, result in substantial energy use and potential reagent losses due to evaporation, whereas a higher reagent concentration leads to higher ash removal. As input parameters (Table 1) for optimizing the iodine number of carbon black, the reflux temperature (A), carbon black/nitric acid ratio (B), and reflux time (C) were chosen from a variety of parameters that affect the iodine number. Seventeen experimental runs based on the Box–Behnken matrix (Table 2) with three center points were conducted to determine the influence of the independent variables on the iodine concentration. The interactive effect of the parameters (temperature (30–50 °C), time (6–24 h), and ratio of carbon black to chemical (0.25–1.0 g/ml)) on the iodine number was studied, and the optimal process parameters were determined using ANOVA. The experimental iodine numbers of carbon black are listed in Table 2. The yields of treated carbon black were in the range of 55%–88%, the yields varied because carbon black loosed by washing step. The experimental data were imported into the software for processing, and the quaternion quadratic polynomial equation for characterizing the relationship between the dependent and independent variables was derived using Eq. (2).

$$Iodine\ number, \frac{mg}{g} = 71.222 + 3.06453A - 0.3745B - 4.17556C + 1.0412AB + 0.044783AC - 0.138222BC - 0.049382A^2 - 30.998B^2 + 0.80084C^2 \quad (2)$$

Considering Eq. (2), the three investigated variables had a linear and quadratic impact on the iodine number. A positive sign signifies a synergistic effect, whereas a negative sign indicates an antagonistic effect [19]. Using the developed model equation, Figure 2 shows the predicted versus experimental values. Variance analysis of the fitted model was conducted. Based on the data in Table 3 for the model, $F = 61.13$, $P = 0.0001$, $R^2 = 0.9874$, and $adj.\ R^2 = 0.9713$, with a lack-of-fit term $p > 0.05$, which was not statistically significant. The Prob > F value indicated an insignificant effect of the 'ratio and time' on the iodine number, whereas the variable 'temperature interaction with ratio and time' was significant because the p-value was less than 0.05, whereas the variable 'ratio interaction with time' was not significant because the p-value was higher than 0.05. The percentage contribution of each parameter indicates that the reflux temperature is the most significant factor

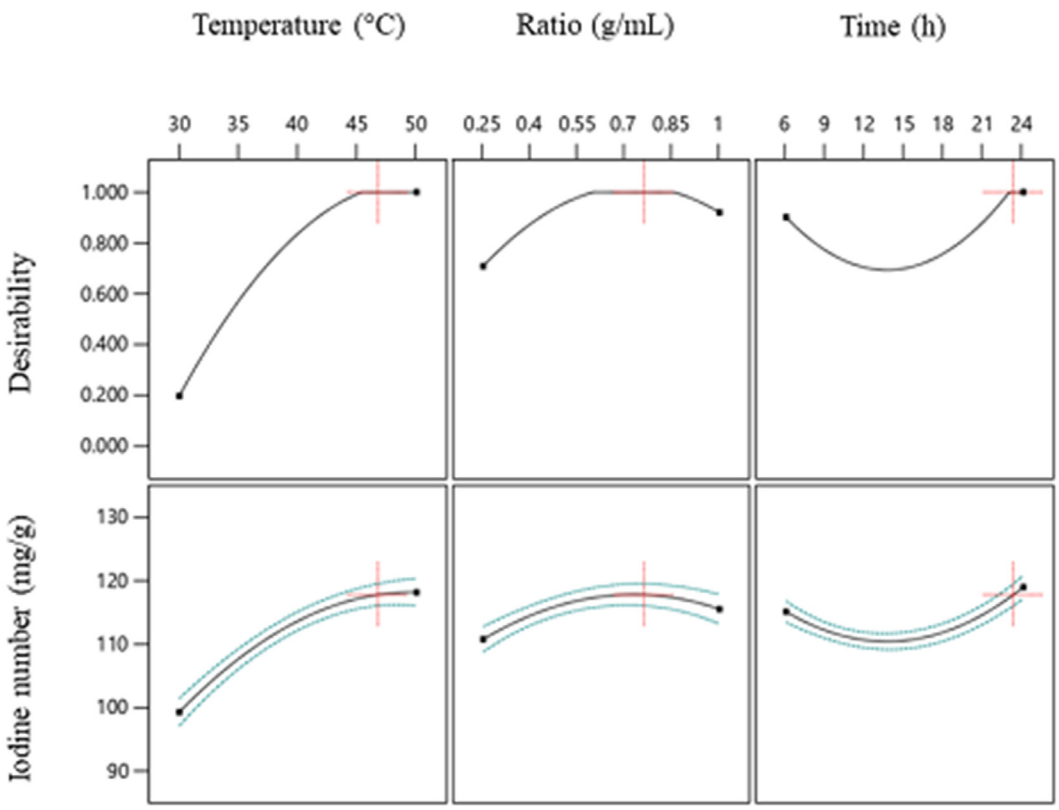

Figure 7. Optimization plot for iodine number.

Table 4. Heavy metal content of carbon black.

	Element (ppm)								
Sample	S	Cl	K	Ca	Mn	Fe	Co	Cu	Zn
RCB	1.6484	177	657	2856	10.06	1386	239	158	14871
RCB_13	0.7889	109	441	69.1	2.94	213	13.69	10.66	641
RBC_EQ	0.9351	81.49	43.45	31.57	0.87	28.7	5.08	12.76	253

contributing to the iodine number. This reflux temperature demonstrates a 24.14% contribution to the iodine number of RCB from the pyrolysis plant.

3.1.2. *Effect of chemical treatment on the iodine number of carbon black*

The three-dimensional response surface plot (Figure 3) illustrates the effect of the interaction between the A and B on the iodine number. A was varied from 30 to 50 °C; B was varied from 0.25 to 1 g/mL, and C was maintained at an intermediate level (12 h). Increasing factors A and B at a low level (0.25 g/mL) significantly increased the iodine number to a certain limit at 40 °C, resulting in the highest iodine number (97 mg/g). However, increasing parameter B while maintaining parameter A as constant could shift the iodine concentration beyond 97 mg/g (approximately 102 mg/g). This 3D surface plot of combination effects between temperature and ratio, the iodine number increased with the rise in temperature and ratio.

Figure 4 shows the effect of the interaction between the A and reaction time (C) on the iodine number, when B was maintained at 0.5 g/mL. At the maximum levels of both A and C, the iodine concentration was maximal (117 mg/g), which may not be optimal considering the increasing trend of factor B. The amount of iodine decreased to 112 mg/g when factor C was decreased to its lowest level (6 h) while maintaining factor A at its highest level (50 °C).

Figure 5 shows the effect of the interaction between B and C on the iodine number, when A was maintained at 40 °C. Upon varying factor B, the iodine concentration reached 0.7 g/ml, whereas the highest concentration was observed when factor C was 24 h. In contrast, the minimum iodine number was achieved at the lowest levels of parameters B and C. This combination effect seems not significant to the iodine number.

Numerous studies have reported the use of other chemical reagents for the demineralization of RCBs. By varying the soaking time at a very low reagent concentration and a low RCB/reagent ratio, the surface area increased with the soaking time [7, 21, 22, 34]. As high temperatures have a substantial impact on energy expenditures and the potential for reagent loss through evaporation, they are a critical factor in the solubilization process. The ratio of RCB to reagent is critical since it is linked directly to the reagent quantity necessary to facilitate the process of demineralization. The effect upon demineralization is increased with greater reagent concentrations and volumes [7] along with the resulting increase in surface area.

3.1.3. *Optimization of chemical treatment process parameters*

Utilizing the software Design Expert's numerical optimization technique, the optimal conditions for maximizing the iodine number were determined based on the Box–Behnken design of the RSM. Desirability function analysis was simultaneously employed in the optimization procedure to obtain the maximum iodine number and optimize the variables for producing carbon black from waste tire pyrolysis [26] (Figure 6). The optimized D value was 1.00. In summary, it is accepted

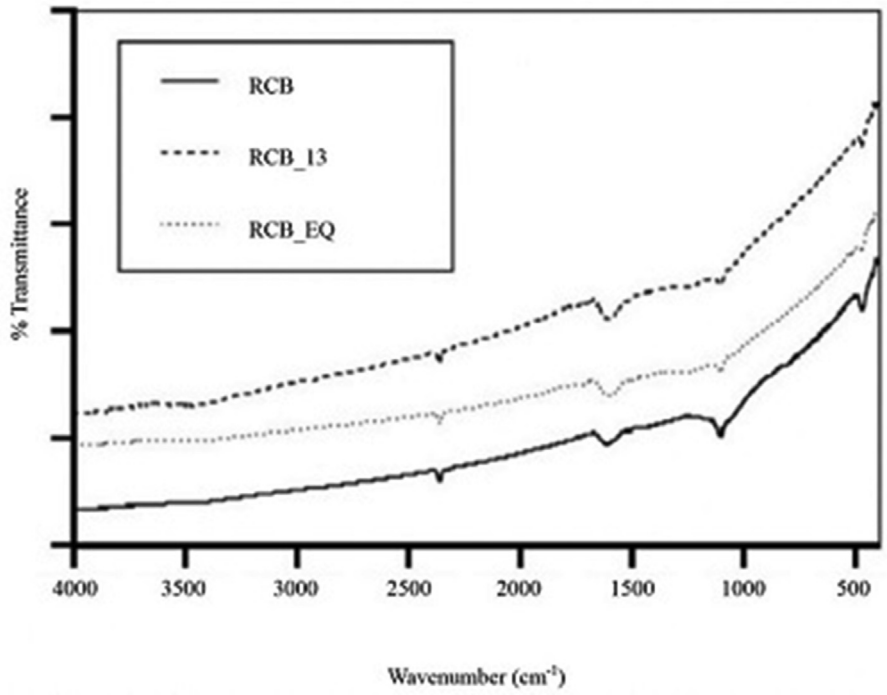

Figure 8. FTIR spectra of carbon black.

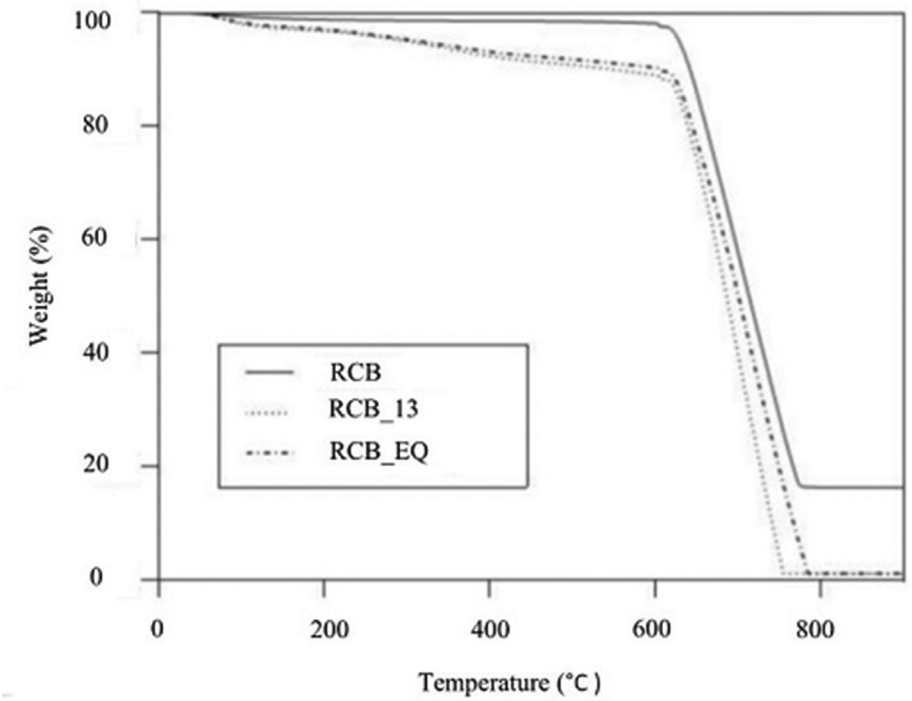

Figure 9. TGA curves of carbon black.

that this is a global solution. For carbon black, the maximum desirability values are: 1.0 for iodine number, between high values of temperature and mean values of ratio can be seen in Figure 6(a) while the time to achieve the highest desirability at the high temperature can be seen in Figure 6(b) and finally in Figure 6(c) is the desirability in the relationship between time and ratio at 40 °C. It was found that the average ratio gave the lowest desirability. And the results from the RSM optimizer shown in Figure 7, the predicted a maximum iodine number of 117.34 mg/g at optimum parameter values of 46.74 °C (A), 23.24 h (C), and 0.76 g/mL (B). The software prediction was validated by conducting additional experiments under the specified conditions; the iodine number was determined to be 119.12 mg/g, with 1.49% error.

3.2. Demineralization

During manufacturing, a number of heavy metals, including calcium, zinc, and iron, can be added to the tires. The content of all metals in tire-derived activated carbon was reduced compared to that in tire char [27]. These inorganic substances are present in RCB because they are not volatilized at conventional pyrolysis temperatures [7]. Consequently, the ash (CaO, Fe_2O_3, ZnO, SiO_2, and K_2O) concentration in carbon black obtained via waste tire pyrolysis is higher than that of CCB [26]. The XRF data for RCB, RCB_13, and RCB_EQ are presented in Table 4. RCB is produced through the pyrolysis of used tires; the proportion of ash, which is predominantly composed of inorganic components, increased by demineralization [27, 28]. When the ash composition underwent analysis via XRF, it became apparent that pyrolysis char contained inorganic compounds mostly comprising Si, Ca, and Zn [29], whereas Zn, Ca, and Fe were detected in high concentrations in RCB. Carbon black is generally treated using an acid demineralization process to reduce organic impurities and eliminate undesirable components [30]. Almost all inorganic impurities, such as zinc, were eliminated via HNO_3 treatment (decreased by 98.29%), which could be anticipated since HNO_3 serves to produce $ZnNO_3$ as a soluble salt when the zinc derivatives are transformed [31]. RCB_13 (Run no. 13) displayed the optimal results in the experiments (the highest iodine concentration), while RCB_EQ was optimized based on surface response methodology. Contents of most heavy metals decreased by more than 90%, whereas the sulfur and chlorine contents decreased by 43.27% and 53.96%, respectively.

3.3. Surface functional groups

Figure 8 displays the FTIR spectra of carbon black. The absorption peaks which are characteristic of RCB_13 and RCB_EQ were considerably similar. However, in contrast with RCB, the peak at 2357 cm^{-1} was attributed to C=O stretching [10] and the peak at 1099 cm^{-1} was attributed to S=O-containing functionalities, including sulfonamides, sulfones, and sulfoxides [32]. The FTIR spectra of RCB_13 and RBC_EQ exhibited a peak at 1586 and 1587 cm^{-1}, respectively, in the region between 1800 and 1500 cm^{-1}, indicative of changes during thermal oxidation [33]; however, peaks of S=O were not detected owing to demineralization. The absorption bands at 1615, 1586, and 1586 cm^{-1} for RCB, RCB_EQ, and RCB_13, respectively, correspond to C=C stretching in the aromatic compounds [14, 33, 34].

3.4. TGA

The TGA profiles of RCB, RCB_13, and RCB_EQ are shown in Figure 9. In the initial stage up to 100 °C, less than 5% moisture removal was observed in all cases. The thermal decomposition of RCB started at 620–770 °C, with an ash content of ~17%. A small variation in the weight loss was observed for RCB_13 and RCB_EQ when the temperature was increased to 620 °C. However, when the temperature exceeded 620 °C, the weight of RCB_13 and RCB_EQ decreased significantly compared to that of RCB, indicating that demineralization has the effect of removing the minerals from the activated carbon along with the inorganic residues. When the temperature is in the range of 620 °C to around 800 °C, a significant loss of weight becomes apparent as a consequence of the removal of the carbon black additive from RCB [35]. The weight loss pattern of RCB 13 was comparable to that of RCB EQ. However, the weight loss of RCB 13 and RCB EQ was higher, probably as a consequence of the oxygen functional groups accumulated as demineralization took place.

4. Conclusions

The demineralization of RCB from a waste tire pyrolysis plant using HNO_3 increased its surface area, as indicated by iodine number measurements, upgrading RCB to commercial-grade N330. Temperature (30–50 °C), time (6–24 h), and the ratio of carbon black to chemical (0.25–1 g/mL) were investigated as variables. Using ANOVA, the significant factors influencing each experimental design response were identified. The quadratic model was statistically significant. The R^2 value of 0.9874 validated the model fit. Treatment at 46.74 °C for 23.24 h with a B of 0.76 g/mL was optimal, as validated by the equation, for increasing the iodine number from 34.4 to 119.12 mg/g. The content of most heavy metals decreased by more than 90%, whereas the sulfur and chlorine content decreased by 43.27% and 53.96%, respectively. The RCB_13 carbon black additive was eliminated at temperatures between 620 and 800 °C, as determined via TGA. The reduced intensity of the peak of S=O-containing functionalities indicates the oxidation of carbon black.

Declarations

Author contribution statement

Natthawat Thonglhueng: Performed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Rinlada Sirisangsawang: Conceived and designed the experiments; analysis tools or data.

Somboon Sukpancharoen: Conceived and designed the experiments; Analyzed and interpreted the data.

Natacha Phetyim: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data included in article/supp. material/referenced in article.

Declaration of interest's statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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