

An investigation into the preparation and characteristics of carbonized rice straw for black food dye

Erni Sofia Murtini^a , Hendrix Yulis Setyawan^b , Sudarminto Setyo Yuwono^a ,
Luluk Mamluhah^c, Lisa Fitri Rahayu^a, Mingming Zhu^d  and Chiemeka Onyeka Okoye^e 

^aDepartment of Food Technology, Universitas Brawijaya Veteran St, Malang, East Java, Indonesia; ^bDepartment of Agroindustrial Technology, Universitas Brawijaya Veteran St, Malang, East Java, Indonesia; ^cIntegrated Laboratory, Universitas Brawijaya Veteran St, Malang, East Java, Indonesia; ^dSchool of Water, Energy and Environment, Cranfield University, Cranfield, Bedfordshire, UK; ^eCentre for Energy (M473), The University of Western Australia, Crawley, Australia

ABSTRACT

Traditional natural dye sources have limitations in terms of variability and availability. This study investigate explored rice straw as a sustainable food dyes, focusing on carbonised rice straw (CRS). The aim was to reduce the CRS silica content to meet the regulatory standards without compromising the dye quality. Various treatments with Sonication, HCl, NaOH, and pressurised NaOH effectively reduced silica to 0.96%, accompanied by increased sodium content. Optimisation of the NaOH concentration seeks to balance the silica reduction and sodium levels. Additionally, the impact of sonication on CRS particle size was assessed to enhance dye stability. The results showed that sonication effectively reduced CRS particle size, with longer durations and higher amplitudes leading to smaller particles and decreased sedimentation rates, indicating enhanced suspension stability. Optimised sonication conditions produced a liquid dye with desirable traits, including uniform particle distribution and minimal sedimentation at 9.0%. This study highlights the potential of rice straw as a sustainable and cost-effective alternative for black food dye production. By refining silica reduction and sonication processing techniques, CRS-derived black food dyes can meet quality standards, reduce agricultural waste, and support sustainable food production.

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1. Introduction

The visual appeal of a food product is the primary indicator of its perceived quality, rather than taste, touch, and smell, and is often correlated with the sense of colour (Spence, 2019). For this reason, yellow (Pranowo et al., 2019) or black-coloured food (Murtini et al., 2018) has become a trend, as proven by many products such as bread, pizza, ice cream, noodles, and cakes. The commercial black dye in the market is vegetable carbon obtained through extraction, isolation, or derivatisation processes from plants (e.g. coconut shell charcoal and bamboo charcoal) or other natural sources. Black food dye is categorised as a natural dye derived from vegetable carbon (CI. 77266). Traditionally, sources of natural black dyes have been limited, largely because of the scarcity of natural resources that can produce a stable, intense black colour suitable for food applications. Natural sources, such as squid ink, black carrot, and certain berries, are either expensive, seasonally available, or yield varying shades that can be unsatisfactory for consistent food production. Considering these challenges, there is a pressing need to explore alternative sources of black food dyes that are not only cost-effective but also readily available, sustainable, and capable of producing a consistent colour quality. Additionally, potential contamination by polycyclic aromatic hydrocarbons (PAHs) formed during pyrolysis warrants investigation to ensure food-grade safety.

CONTACT Hendrix Yulis Setyawan  hendrix@ub.ac.id  Department of Agroindustrial Technology, Universitas Brawijaya Veteran St, Malang, East Java, Indonesia

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One such promising resource is rice straw, a byproduct of rice cultivation, which is abundantly available globally, especially in regions where rice is a staple crop. Conventionally, agricultural waste, which is often burned, contributes to air pollution, and it holds potential as a valuable raw material. Indonesia has abundant rice, producing over 54 million tons in 2022, representing approximately 10% of the global rice production (FAOSTAT, 2024). However, the straws are either dumped in the landfill or used as animal feed, which is undesirable. Straw, like other biomass, can be thermally pyrolysed to produce activated carbon for adsorbent (Ghaedi et al., 2011, 2012, 2016) and carbonised rice straw (CRS) for black food dye (Murtini et al., 2019). However, the silica content in rice straw is 30–60%, which is too high to meet the standard of 2% (FDA, 2018).

The silica content can be reduced by various methods, such as conventional silica reaction with Na_2CO_3 at 1400°C (Todkar et al., 2016), superheated steam, leaching by acid solvents (Navaee-Ardech et al., 2003) and alkaline solvents (Mittal, 1997). Mittal (1997) treated CRS with HCL 12N, alkaline (NaOH 40%), and pressurised alkaline (NaOH 40%) and showed that treatment with pressurised alkaline resulted in a significant reduction in silica to 0.96% and an increase in carbon content to 75.1%. NaOH 40%, however, increased the Na content of the CRS to 3.6%.

In other words, reducing the silica content entails a trade-off with the sodium content. Therefore, further research on process optimisation is necessary to reduce silica levels without elevating sodium concentrations. Similarly, the black food dye of vegetable carbon is usually in the form of a viscous liquid by suspending the black food dye (approximately 12%w/w) in glucose syrup, glycerine, or propylene glycol (MacDougall, 2002). The particle size of CRS in black food dye should be less than 5 µm to suppress the sedimentation of carbon. Sonication has been found to produce a finer CRS particle size and a more uniform dispersion of CRS in a viscous liquid, thereby hindering sedimentation (Wang et al., 2022). Comparative performance testing against a commercial vegetable carbon dye (E153) in model food systems is essential to demonstrate industrial relevance. Hence, the present research aims to close these gaps by (i) preparing CRS-based food dyes using waste rice straw and (ii) evaluating the effects of NaOH concentration, pressure, and sonication on the silica content, particle size of CRS, and stability of the prepared food dye. The anticipated outcome of this research is expected to advance environmental sustainability and cleaner production through the utilisation of agricultural by-products, thereby reducing waste while satisfying the requirements of contemporary food production. This work represents the first systematic evaluation of CRS as a food-grade black colourant, integrating safety assessments, process optimisation, and comparative performance analysis.

2. Experimental

2.1. Materials

The material used in this study include rice straw (leafless tops of black rice straw, sourced from farmers in Trawas, East Java, Indonesia); Hydrochloric acid (HCl, 37%; ACS reagent grade); Sodium hydroxide (NaOH, ≥99% purity, food-grade), and high-fructose syrup (HFS, ≥55% fructose, food-grade) were purchased from Merck (Darmstadt, Germany); Acetonitrile (HPLC grade); sodium chloride; anhydrous magnesium sulphate; and primary/secondary amine (PSA) for QuEChERS were procured from Sigma-Aldrich (St. Louis, MO, USA).

2.2. Apparatus

The apparatus are: pyrolysis reactor - an 80 cm vertical fixed-bed quartz reactor with 3500W furnace (up to 800°C, Carbolite Gero, Germany); Planetary ball mill: PQ-N04 series (100rpm, Pulverisette, Fritsch GmbH, Germany); Sieves: ASTM #40, #100, #200, #350 (stacked); pH metre: Ezdo PL-600 (Ezdo, China); Colour reader: Minolta CR-100 (Konica Minolta, Japan); Particle size analyser: Cilas 1090 (Malvern Panalytical, UK); Autoclave: GEA (GEA Group, Germany); SEM: Hitachi S-3000 (Hitachi, Japan); BET surface area analyser: BSD-PM (Micromeritics, USA); LC-MS/MS & GC-MS/MS: triple quadrupole system (Thermo Fisher Scientific, USA); Magnetic stirrer: MS-H-Pro GSA (IKA, Germany). Ultrasonic probe: Cole Parmer WB400 (20 kHz, 400W, Cole-Parmer, USA).

2.3. CRS preparation

The raw material used was the leafless top of black rice straw from farmers in Trawas, East Java, Indonesia. Rice straw was carbonised in nitrogen at 550°C, to maximise carbon yield while minimising PAH formation, in pyrolysis reactor (up to 800°C) to produce carbonised rice straw (CRS). The CRS was then crushed in planetary ball mill at 100rpm for 120min and sieved gradually through 40, 100, 200, and #350 meshes.

The physical characteristics of the raw materials used are listed in Table 1. The CRS was further analysed for its colour, pH, and particle size. Figure 1 shows that the CRS colour is #41403b colour RGB value is (65,64,59)–dark grey to black. The colour analysis result showed that CRS had an L^* value of 26.8 ± 0.02 , a value of -2.1 ± 0.03 , and a value of 3.2 ± 0.03 . The black colour is due to the carbon component in the CRS and the greyish colour is silica. According to Martini et al. (2019), the black colour lies in CRS, ranging from 7% to 44%, depending on the rice varieties and the part of rice straw used in CRS making.

The particle size of CRS was D90 104.6 μm , D50 35.1 μm , and D10 4.2 μm . The particle size of the CRS produced in this study was smaller than that reported by Murtini et al. (2019) because of the ball-mill milling process. The particle size reduction process occurs in the ball mill by transferring the kinetic energy of the moving grinding media to the milled sample (Monov et al., 2012).

The pH of the CRS was 10.6 ± 0.02 . CRS has pH values ranging from 10–11 depending on the rice variety and the part of rice straw used in CRS making (Murtini et al., 2019). CRS is alkaline, with mineral components ranging from 42 to 87%. Approximately 11–27% of the minerals in CRS are in alkaline and alkaline soil groups, such as Ca, Mg, K, and Na. The pH of CRS is higher than that of bamboo charcoal reported by Zhenchao et al. (2015) at pH 9.7. Furthermore, the pH value of CRS is stable during storage, as demonstrated by a constant pH value during 15-day storage at 40°C under direct light exposure (Murtini et al., 2019).

The straw and CRS were analysed for their pesticide residue content (Table 2), such as cypermethrin, cypermethrin alpha, abamectin, and 2,4D, using HPLC with a triple quadrupole Tandem Mass Spectrometry detector (LC-MS/MS) following the QuEChERS extraction method (Nuapia et al., 2016). In brief, a 10g sample of homogenised food was placed into a 50ml Teflon tube, followed by the addition of 10ml of acetonitrile, and the mixture was vigorously shaken for 1 min. The salting-out process was conducted by adding 1.5g of sodium chloride and 3g of anhydrous magnesium sulphate to the tube, shaking

Table 1. Raw material characteristics of carbonised rice straw (CRS).

Parameter	Analysis result	Literature
Colour		
(L^*)	26.8 ± 0.02	28.97 ± 0.06^b
(a)	-2.1 ± 0.03	7.38 ± 0.03^b
(b)	3.2 ± 0.03	5.32 ± 0.05^b
Particle size		
D90 (μm)	104.62 ± 0.94	114.13^b
D50 (μm)	35.09 ± 0.02	49.13^b
D10 (μm)	4.16 ± 0.04	12.76^a
pH	10.62 ± 0.02	10.53 ± 0.03^b

Description: The data are presented as the average of three repetitions; the numbers following $\pm$ are standard errors.

Source:

^a= Murtini et al. (2018).

^b= Murtini et al. (2019).



Figure 1. CRS colour interpretation (#41403b).

Table 2. Pesticide residue analysis on the rice straw in carbonised rice straw (CRS).

Type of pesticide	Limit detection (ppm=mg/kg)	Rice straw	CRS
2,4 D	0.050	Nd	nd
Abamectin	0.010	Nd	nd
Alpha-cypermethrin	0.010	–	nd
Cypermethrin	0.010	0.134	nd

vigorously for 1 min, and then undergoing centrifugation. After centrifugation, the clear supernatant (6.5 mL) was transferred to a polypropylene tube for clean-up using ~1.6 g of anhydrous magnesium sulphate and 27.5 mg of primary/secondary amine (PSA). The solution was re-centrifuged and injected into the LC-MS/MS and GC MS/MS systems.

2.4. Reduction of silica content

CRS was washed using HCl, NaOH, and NaOH under high pressure to reduce silica, following the method of Todkar et al. (2016). First, the silica content was reduced by dissolving 50 g of CRS in 500 ml HCl 12.06 N. The samples were then homogenised using a hot plate stirrer at 700 rpm for 30 min and filtered. The filtered CRS residue was washed with distilled water until a neutral pH was achieved and then dried in an oven at a temperature of 105 °C for 24 h. Second, silica was reduced by dissolving 50 g of CRS in 500 ml of 40% NaOH. The samples were then homogenised using a hot plate stirrer at 700 rpm for 30 min and filtered using filter paper. The filtered CRS residue was washed with distilled water until a neutral pH was achieved and then dried in an oven at a temperature of 105 °C for 24 h.

Third, the silica was reduced by washing with NaOH under pressure. CRS (50 g) was dissolved in 500 ml NaOH. The NaOH variations used in the study, including the different concentrations (5%, 10, 15, 20, 25, 30, 35, and 40%). The mixture was homogenised using a magnetic stirrer (MS-H-Pro GSA) at 700 rpm for 15 min and then placed in an autoclave at 1.48 atm (0.15 Mpa) and 120 °C for 15 min. It was subsequently sieved using filter paper. The residue left on the filter paper was washed with distilled water until it reached a neutral pH before it was placed in an oven at 105 °C for 24 h.

All treatments were repeated three times. Three CRS structures with or without treatment were observed with an accelerating voltage of 0.5–30 kV and a secondary electron resolution of 0.8 nm (accelerating voltage of 15 kV). Nitrogen adsorption and desorption were performed at –196 °C. The sample was degassed at 250 °C for 180 min, and the specific surface area was calculated.

The carbon and mineral contents of the CRS samples were measured. The data were analysed using ANOVA with a 95% confidence interval and tested using Tukey's 95% confidence interval. Data are presented as mean ± standard error (SE). The treatment that produced CRS with the lowest silica and Na content was considered the best treatment, and it will be utilised in liquid dye production.

2.5. Liquid dye production

The liquid dye production followed the approach of Douglass (2002). CRS (10 g) was mixed with 100 ml high fructose syrup (HFS), which was homogenised using a magnetic stirrer at 700 rpm for 30 min. The mixture of CRS and HFS was then sonicated using a 20 kHz ultrasonic frequency ultrasonic probe at 40, 50, and 60% amplitude. Sonication was performed for 60, 90, and 120 minutes. Each treatment was repeated five times. The liquid dye was analysed for colour, settling rate, and particle-size distribution (D10, D50, and D90). The data were analysed using an analysis of variance (ANOVA) 95% confidence interval and Tukey's 95% confidence interval. Data are presented as mean ± standard error (SE).

2.6. Characterization

The mineral content of CRS was measured using X-ray fluorescence (XRF). The total carbon, organic, and inorganic carbon of the CRS were measured using a total combustion analyser.

The particle size distribution of CRS was measured using a particle size analyser (Cilas 1090). Each particle was grouped into three sizes: D10, D50, and D90. The colour of the liquid dye was measured.

The pH was measured using a pH metre. The settling percentage was measured by placing the liquid dye into a measuring cylinder, then sizing its initial volume, leaving it for 48 h, and measuring the sedimentation volume formed. The formula used to measure the settling percentage is the sediment volume divided by the initial volume and multiplied by 100%.

3. Results and discussion

3.1. Reduction of silica using HCL, NaOH 40% and pressurised NaOH 40%

Treatment with HCl 12.06 N, NaOH 40%, and NaOH 40% at ~1.5 atm was used to reduce the silica content of the CRS. The control was raw CRS that was not treated with the solvent. Table 3 shows that the silica content of the CRS control is 29.9%. Treatment with HCl 12.06 N increased the proportion of silica content of CRS to $34.5 \pm 0.05\%$ due to the reduction of several mineral components, such as iron, calcium, and potassium, which reacted with HCl. The reaction of HCl with several components (Equations 1–3) shows that the minerals in CRS reacted with HCl to form a salt (Bi et al., 2005). PAH analysis confirmed levels below detection limits (<0.1 mg/kg), ensuring safety of acid and base treatments. The salt was dissolved in distilled water during filtering. According to Haynes (2011), FeCl_3 is a water-soluble salt (with a solubility of 92 grams/100 ml at a temperature of 20°C), and its solubility increases with temperature. Calcium chloride (CaCl_2) is also water-soluble, yet it has a lower solubility, viz. 75 gram/100 ml at a temperature of 20°C , and its solubility increases with temperature (Vrana, 2001). The water solubility of potassium chloride (KCl) is 74.5 grams/100 ml at 20°C (Pinho & Macedo, 2005). The more mineral components lost, the more silica components were detected by XRF.



Table 3 shows a decrease in the detected silica with NaOH 40% to $\sim 3.7 \pm 0.16\%$ and NaOH 40% with a pressure of 1.5 atm to $\sim 1 \pm 0.00\%$. The reaction of silica and NaOH forms sodium silicate, which can be separated from the CRS in the filtering process (Nandiyanto et al., 2017). The residual sodium silicate in CRS was removed in the neutralisation process using distilled water because it has a good water solubility of 22.2 gram/100 ml at a temperature of 20°C and increased to 160 gram/100 ml at 80°C (ILO, 2017). The treatment that produced the lowest silica content was the CRS treatment using NaOH 25% at

Table 3. The effects of HCl 12.06 N, NaOH 40%, and NaOH 40% at 1.48 atm on the mineral component.

Mineral component	Treatment				Literature ^a
	Control	HCl 12.06 N	NaOH 40%	NaOH 40% at 1.48 atm	
SiO_2 (%)	29.88 ± 0.00	34.50 ± 0.05	3.66 ± 0.16	0.96 ± 0.00	42.51 ± 0.06
Al_2O_3 (%)	nd	nd	nd	nd	nd
Fe_2O_3 (%)	0.37 ± 0.00	0.04 ± 0.00	0.27 ± 0.01	0.41 ± 0.02	0.12 ± 0.01
MnO (%)	0.100 ± 0.00	nd	0.070 ± 0.00	0.140 ± 0.00	0.090 ± 0.00
CaO (%)	0.47 ± 0.00	0.08 ± 0.00	0.60 ± 0.00	0.64 ± 0.00	0.80 ± 0.00
MgO (%)	0.34 ± 0.00	0.03 ± 0.00	0.57 ± 0.00	0.70 ± 0.00	0.62 ± 0.01
K_2O (%)	8.335 ± 0.00	0.130 ± 0.00	0.323 ± 0.00	0.240 ± 0.00	17.160 ± 0.01
Na_2O (%)	0.03 ± 0.00	0.01 ± 0.00	1.50 ± 0.16	3.55 ± 0.01	0.71 ± 0.00
P_2O_5 (%)	0.730 ± 0.00	0.080 ± 0.00	0.187 ± 0.00	0.195 ± 0.00	0.32 ± 0.00
SO_3 (%)	0.385 ± 0.00	0.080 ± 0.00	0.093 ± 0.02	0.115 ± 0.00	0.32 ± 0.00
TiO_2 (%)	nd	nd	nd	nd	0.01 ± 0.00
V_2O_5 (%)	nd	nd	nd	nd	nd
BaO (%)	nd	nd	nd	nd	nd
ZnO (%)	0.020 ± 0.00	0.010 ± 0.00	0.010 ± 0.00	0.010 ± 0.00	0.02 ± 0.00
Total C (%)	43.50 ± 0.05	47.36 ± 0.08	70.10 ± 0.20	75.05 ± 1.75	nd

The data presented are the average of three replicates; the numbers following $\pm$ are standard errors. The pressure presented is *gauge* pressure. The control group was the untreated CRS group.

Source: a=Murtini, et al. (2018).

elevated pressure (~1.5 atm). The elevated pressure increased the reaction temperature, resulting in increased collisions between the molecules in the solution to accelerate the reaction. Moreover, the solubility of silica in water increases with increasing pressure and temperature increase (Icopini et al., 2005).

CRS structures with and without acid and base treatments are shown in Figure 2. The addition of pressure made the CRS structure finer and caused more silica to react with NaOH, producing CRS with a lower silica content. A treatment to reduce the silica level by reacting CRS with HCl, NaOH, and pressurised NaOH also affected the CRS surface area as measured by BET. SEM micrographs (Figure 2) show removal of surface silica clusters and increased porosity after pressurised NaOH treatment, correlating with the 22–28× increase in BET surface area (Table 4). One of the purposes of the CRS test is to estimate the rate of dissolution. Theoretically, the greater the surface area, the higher the probability of interacting with water. The CRS surface area and porosity measurement results for the various treatments are presented in Table 4, showing the increase in the CRS surface area by 22-to 28-fold following treatment with HCl and NaOH. This increase occurred because the minerals were reduced, and the carbon content increased after the treatment (please see mineral and carbon data in Table 3). Untreated CRS

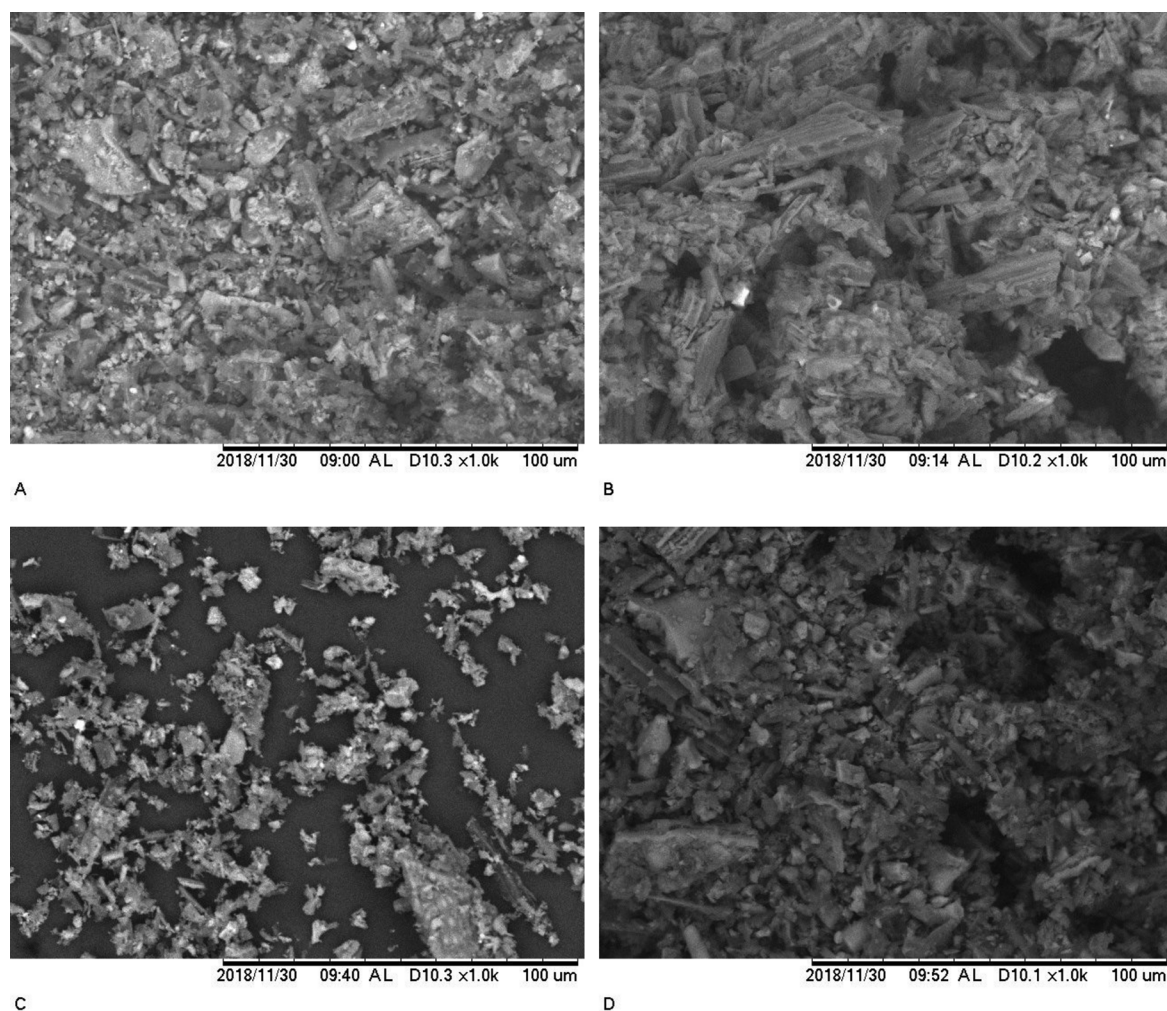


Figure 2. CRS structure with and without treatment for reducing silica seen by SEM under 1000 x magnification (A) Without treatment, (B) NaOH 40%, (C) NaOH 40% at 1.48 atm, (HCl 12.06 N).

Table 4. Carbonised rice straw (CRS) surface area and pore size in the various treatments.

Treatment CRS sample	BET surface area (m ² /g)	Pore size diameter (nm)	Pore size width (nm)
Untreated CRS	6.48	3.87	35.04
NaOH 40%	185.96	2.74	3.31
Pressurised NaOH 40%	145.84	2.95	3.71
HCl	156.62	2.11	2.34

seems to have a low surface area owing to its high mineral content, similar to other biochar (Setyawan et al., 2024). Treatment with NaOH under elevated pressure is useful. However, the use of 40% NaOH caused an increase in CRS's sodium content of the CRS. However, this residual Na remains below WHO food additive limits (max 5%). Further research is required to reduce the NaOH concentration used in the purification process.

3.2. Effect of NaOH addition on CRS characteristics

NaOH addition at various concentrations was performed to determine the high silica reduction with a tolerable sodium content. The silica, sodium, and carbon contents of CRS treated with pressurised NaOH at various concentrations are listed in Table 5.

3.2.1. Silica content

Table 5 shows increasing NaOH concentration reduces silica content in the CRS, but there was an increase in NaOH by 30 to 40%. The lower silica content produced by treatment with a NaOH concentration of 25% amounted to $1.1 \pm 0.01\%$. Selecting 25% NaOH provides silica $<2\%$ and Na $<2\%$, meeting EFSA/FDA guidelines. The decrease in silica levels occurred due to the reaction between silica and NaOH that formed sodium silicate (Todkar et al., 2016). The increasing NaOH concentration used causes an increase in sodium silicate formation, resulting in more optimal silica extraction. Furthermore, if the NaOH concentration used was higher, then the solvent's contact with the area of the material increases. Accordingly, the solvent distribution to the husk ash is also higher. Solvent distribution in the husk ash caused an increase in sodium silicate forming and yield silicate (Rhevi et al. 2014). The sodium silicate formed will be separated from the CRS through a filtering process to produce a low silica CRS. The remaining sodium silicate in the CRS will be dissolved using distilled water in the neutralisation process.

The increase in the extracted silica level owing to the increase in NaOH concentration was also reported by Setyawan et al. (2019). Extraction of rice husk ash using NaOH 0.5N, 0.75 N, and 1 N solvent produced silica yield by ratio 1:6 (materials: solvent) with the following concentrations: NaOH 0.5 N was 16.3%, NaOH 0.75 N was 41.3%, and NaOH 1 N was 62.8%.

The research results showed that at NaOH concentrations of 30, 35, and 40%, the silica content was greater than that at NaOH concentrations of 20% and 25%. The greater the NaOH concentration, the more sodium silicate was formed. On the contrary, increasing the NaOH concentration from 30 to 40% caused saturation of NaOH and reduced its silicate dissolving ability. Based on these results, CRS's silica content of CRS has been successfully reduced to the FDA limits (FDA, 2018).

3.2.2. sodium content

Table 5 shows that the addition of NaOH increased the sodium content in CRS, which ranged between ~ 1.9 and 3.0% . This sodium content is low for food dyes used in cooking, considering the maximum daily sodium intake of 2000mg/day. The higher the NaOH concentration, the higher the sodium content in the CRS. The lowest sodium content was observed in the 5% NaOH treatment, and the highest was observed in the 40% NaOH treatment. The increase in sodium content was caused by a reaction between NaOH and several other mineral components of CRS, viz., Fe_2O_3 producing $\text{Fe}(\text{OH})_3$, CaO producing $\text{Ca}(\text{OH})_2$ and K_2O producing KOH . These reactions also form Na_2O . The higher the NaOH concentration,

Table 5. The effect of NaOH concentration on the silica, sodium and carbon.

Concentration NaOH (%)	Silica content (%)	Sodium content (%)	Total carbon (%)
5	2.01 ± 0.01^a	1.89 ± 0.01^e	45.77 ± 2.09^d
10	1.22 ± 0.04^b	2.10 ± 0.01^d	48.80 ± 1.19^{cd}
15	1.17 ± 0.00^b	2.27 ± 0.01^c	50.30 ± 1.55^{cd}
20	1.08 ± 0.00^c	2.28 ± 0.02^c	59.17 ± 1.89^{ab}
25	1.04 ± 0.00^d	2.34 ± 0.00^c	65.90 ± 0.65^a
30	1.16 ± 0.00^b	2.70 ± 0.01^b	52.23 ± 0.96^{cd}
35	1.17 ± 0.00^b	2.66 ± 0.01^b	53.30 ± 1.73^{bc}
40	1.16 ± 0.00^b	2.91 ± 0.01^a	50.40 ± 0.37^{cd}

Description: The data are presented as the average of three repetitions; the numbers following $\pm$ are standard errors. Numbers with different letters indicate significant differences ($\bar{I} \pm 0.05$). The reaction was performed at a pressure of 1.48 atm (gauge pressure).

the higher the Na₂O content. The highest sodium content, $2.9 \pm 0.02\%$ was observed in the treatment using 40% NaOH.

3.2.3. total carbon

Table 5 shows that the total carbon of the CRS ranges between 41.6 and 67.2%. The analysis of variance result showed that NaOH concentration has a significant effect ($\alpha=0.05$) on the total carbon content. Total carbon increased with increasing NaOH concentration from 5 to 25% and then decreased from 30 to 40%. The maximum total carbon of $65.9 \pm 0.6\%$ was obtained in the treatment with NaOH 25%. This first systematic study of alkali concentration shows optimal carbon retention at 25% NaOH, balancing mineral removal and organic preservation. While the carbon content is not only the first and most significant CRS component, but also an influential component of the black colour. Carbon is from the organic component left in CRS and is made by an incomplete combustion process. The amount of carbon in each treatment is dependent on reduced silica and other minerals and organic compounds in the CRS. The combustion also produced organic compounds other than carbon, i.e. sulphur. According to Doberman & Fairhurst (2002), the rice straw combustion process removed 5 to 60% sulphur, depending on the burning method used. Sulphur, left in the CRS, can react with NaOH to form Na₂SO₃, which is water-soluble and will mostly be lost during neutralisation. Hydrogen is another component that can react with NaOH and produce sodium hydride (NaH), which has water-soluble characteristics. A reduction of other organic elements, such as sulphur and hydrogen, will affect CRS's total carbon.

3.3. Effect of sonication

The purified CRS was then used as an ingredient in a black food dye made with fructose syrup medium at a CRS: fructose ratio (1:10). Owing to the inert and insoluble carbon characteristics, particle size reduction is required to improve the stability of the dye produced through sonication. This study aimed to determine the effect of sonication duration and amplitude on the colour, particle size, and percentage sedimentation of liquid dye characteristics. The characteristics of the liquid dye at various sonication durations and amplitudes are presented in Table 6.

3.3.1. colour

Table 6 shows that the duration and amplitude of sonication had insignificant effects on the L value. According to Murtini et al. (2019), the black colour of CRS is highly dependent on the number of carbons contained. This research used the same CRS; therefore, the carbon content was the same. The sonication process can cause a change in the particle size; however, it cannot reduce or increase the carbon number. CRS has high colour stability during the storage process, as reported previously. According to Murtini et al. (2019), CRS soaked at 40°C for 15 d under light exposure experienced no changes. Furthermore, the CRS strength of plant-derived natural dyes, such as chlorophyll and anthocyanins, is easily broken down or has low stability. In addition, they are sensitive to external factors, such as temperature, light, pH, and oxygen. Additionally, the stability of light exposure and temperature are the criteria that quality

Table 6. Colour, particle size, and percentage sedimentation characteristics of liquid dye at different sonication duration and amplitude.

Sonication		Particle size					
Duration (min)	Amplitude (%)	Colour L	D10 (μm)	D50 (μm)	D90 (μm)	Uniformity coefficient	% Sedimentation
60	40	25.09 ± 0.04	0.90 ± 0.34	5.96 ± 0.75 ^a	20.59 ± 1.63 ^a	6.62	27 ± 0.01 ^a
60	50	25.14 ± 0.03	0.40 ± 0.23	4.18 ± 0.61 ^b	10.17 ± 0.69 ^{cd}	10.45	21 ± 0.00 ^c
60	60	25.12 ± 0.04	0.10 ± 0.06	3.17 ± 0.07 ^{cd}	6.38 ± 0.11 ^{ef}	13.72	16 ± 0.00 ^d
90	40	25.07 ± 0.04	0.50 ± 0.29	5.11 ± 0.47 ^{ab}	14.48 ± 1.34 ^b	10.22	24 ± 0.00 ^b
90	50	25.00 ± 0.02	0.49 ± 0.28	4.70 ± 0.46 ^b	10.91 ± 1.08 ^{bc}	9.59	21 ± 0.01 ^c
90	60	25.01 ± 0.04	0.02 ± 0.01	2.56 ± 0.10 ^d	4.99 ± 0.14 ^f	12.80	13 ± 0.00 ^e
120	40	24.99 ± 0.03	0.04 ± 0.02	3.36 ± 0.13 ^{cd}	8.57 ± 0.12 ^{de}	8.40	21 ± 0.00 ^c
120	50	25.04 ± 0.02	0.23 ± 0.13	2.56 ± 0.39 ^d	4.78 ± 0.59 ^f	11.13	15 ± 0.00 ^{de}
120	60	24.98 ± 0.02	0.25 ± 0.14	2.30 ± 0.34 ^d	4.09 ± 0.45 ^f	9.20	9 ± 0.00 ^f

Description: The data are presented as the average of three repetitions; the numbers following ± are standard errors. Numbers with different letters indicate significance ($\alpha=0.05$). The percentage sedimentation measurements were carried out after the suspension was left for 48 h.

dyes must possess. The dye stability at high temperatures is crucial because most food processing is carried out at high temperatures, such as boiling, steaming, and drying. Therefore, CRS liquid dye can be applied to food as a natural dye owing to its strength.

3.3.2. Particle size distribution

The particle size distributions of D10, D50, and D90 showed that 10, 50, and 90% of the particle size of the CRS liquid dye were smaller than those listed in Table 6. This observation indicates that the longer the sonication process, the smaller the CRS particle size produced. A strong inverse correlation ($R^2 = 0.92$) between D90 and sedimentation rate confirms the size–stability relationship. The size reduction was affected by the power owing to the cavitation phenomenon that broke the van der Waals bonds between the CRS aggregates (Jung et al., 2012). Cavitation begins with bubble formation, then expands or enlarges the bubbles, and eventually breaks down into smaller bubbles via an explosion (Huang et al., 2012). Arrigo et al. (2018) claimed that in nanocomposite ash making, nanotube ash diameter decreases dramatically from the initial size of 100 nm to 72 nm after sonication for 15 min and continuously decreases with increasing sonication duration. However, after sonication for 120 min, the ash diameter did not change, and 33 nm had the smallest particle size. The effort to produce cavitation strongly correlates with the energy generated during sonication.

The large amplitude is another aspect responsible for the energy produced. The greater the amplitude used in sonication, the greater is the energy produced. Hennrich et al. (2007) stated that the total energy is equivalent to the product of the power utilised and time. An example of the total energy generated by the increase in amplitude is shown by Sabet et al. (2015), in which the nanotube ash was dispersed into the vinyl ester resin using an ultrasonic probe frequency of 20 kHz. The study used amplitudes of 50% and 70% and generated powers (W) of respectively, 35–38 watts and 55–57 watts. Sonication was conducted for 18 min at 50% amplitude, and the total energy produced was 40 kJ. Similarly, it produced 61 kJ at a 70% amplitude within the same duration of the sonication process. The sonication intensity is the energy transmitted (Joule) per unit time (min). The intensity was directly correlated with the amplitude adjustment. Therefore, while a lower amplitude produces a lower sonication intensity, a higher amplitude yields a higher sonication intensity (Alkerabi et al., 2017). The energy generated during sonication induces strong shock waves in the solvent molecules. Shock wave production causes nanotube ash to experience cracks outside, resulting in individual ash separation from its bundle nanotubes or agglomerates (Hilding et al., 2003; Ahir et al., 2008). Loos et al. (2008) stated that nanotube ash dispersion in acetone using sonication at 500 watts of power and a frequency of 20 kHz could increase the solvent viscosity, indicating exfoliation or separation of individual ash from the agglomerates.

Size reduction also occurred during the CRS purification. Silica laid on the CRS surface reacts with sodium hydroxide to form sodium silicate, which leaves porous CRS that can be easily crushed (Nandiyanto et al., 2016). Nanoparticle ash is synthesised through porous ash making by mixing a silica source with an ash source; then, the mixture was heated until it formed solids. After that, the silica was extracted by dissolving the ash into an alkaline solvent to form porous ash, which was used to make the nanoparticle ash.

According to NATCOL (2011), cited in the EFSA. (2012), the particle size of commercial vegetable carbon available on the market has a value of $D_{10} < 2 \mu\text{m}$, $D_{50} < 5 \mu\text{m}$, $D_{90} < 55 \mu\text{m}$, and no particle size below 275 nm (0.275 μm). Therefore, the sonication process can reduce the particle size of CRS in the liquid dye, so that the values of D10, D50, and D90 meet the particle size criteria of commercial vegetable carbon.

The uniformity coefficient was calculated using the distribution curve of the particle distribution. The X-axis denotes the particle size range (μm), and the y-axis denotes the cumulative particles (%). The uniformity coefficient shows the slope of the curve and the uniform properties with respect to the size of the distributed particles. If the coefficient value is low, the curve becomes steeper, and the particle in the CRS liquid dye becomes more uniform, and vice versa. Although there is no standard for a good uniformity value, the obtained results show that the CRS liquid dye has a uniformity coefficient in the range of 6.6–13.7.

Table 7. The characteristics of liquid dye of the best treatment.

Parameter	Value
Particle size of D10 (μm)	0.59 ± 0.14
Particle size of D50 (μm)	2.30 ± 0.20
Particle size of D90 (μm)	4.09 ± 0.45
Percent Sedimentation (%)	9.00 ± 0.00
L* value	24.98 ± 0.02
a (-) value	2.30 ± 0.04
b (+) value	3.60 ± 0.02
pH	7.2 ± 0.1

Description: The data are presented as the average of three repetitions; the numbers following $\pm$ are standard errors. Numbers with different letters indicate significant differences ($l \pm = 0.05$).

3.3.3. Settling percentage

Table 6 shows that the longer the sonication duration, the smaller settling value will be obtained. The largest average of sedimentation was $27 \pm 0.01\%$, provided in the treatment with 60 min of sonication duration. Contrarily, the smallest average of sedimentation was $9 \pm 0.00\%$ in the treatment with 120 min of sonication duration. This observation can be attributed to the decrease in the bond between particles during sonication, which is caused by the cavitation effect that breaks particle bonds (Jung et al., 2012). Therefore, extending the sonication duration results in the breakdown of more particle bonds, which, in turn, leads to decreased particle size and reduced sedimentation rate. Particle size closely relates to the particle's cross-sectional area and the upward pressure of the suspension fluid. The particle size and its cross-sectional area have an inverse relationship.

Nevertheless, a linear relationship was observed between the cross-sectional area and the upward pressure. The larger the particle size, the smaller is the cross-sectional area in the same volume. The larger cross-sectional area of the particles causes the liquid's upward pressure to retard the particle motion and settle even more. Accordingly, particle size reduction was carried out to retard sedimentation, which correlates with the particle size. The data presented in Table 6 show that for the same sonication amplitude, a longer sonication decreases the particle size. The value of D90 at a sonication time of 120 min was smaller than those obtained at sonication times of 90 min and 60 min. Thus, the percentage sedimentation in the sonication for 120 min had the lowest value compared to that in the sonication for 90 min and 60 min.

Table 7 shows that the greater the amplitude used, the lower the percentage of sedimentation of the CRS liquid dye generated. Sonication at an amplitude of 60% produced the smallest sedimentation percentage compared to those at amplitudes of 40% and 50%, indicating that the amplitude affects the energy produced during the sonication process. The greater the amplitude used, the greater the power (W) produced; thus, the total energy becomes even more significant (Henrich et al., 2007). A higher sonication intensity can delay re-agglomeration, resulting in a more stable suspension over a longer time. A high sonication intensity can induce electrostatic repulsion, which can counterbalance the van der Waals tensile forces and produce a more homogeneous and stable suspension. When the high ultrasonic waves passed through the solution, they generated many cavity bubbles that then collapsed and increased the split (break apart) between molecules (Alkerabi et al., 2017). The energy produced during the sonication process can probably break the CRS aggregates formed by Van der Waals bonds between particles (Jung et al., 2012). This is generated by the lower (smaller) particle size of the CRS. The smaller the particle size, the lower the sedimentation rate. According to Stokes' law, the sedimentation rate is directly proportional to particle diameter. The particle size data presented in Table 7 show that the greater the amplitude used simultaneously, the smaller the CRS particle size was getting lower. Moreover, D90, with an amplitude of 60%, has the smallest value compared to those with an amplitude of 50%, and a 40% sedimentation value also decreases.

The best treatment in the CRS liquid dye was determined by the smallest particle size (D10, D50, D90), lowest percentage of sedimentation, and lowest colour (L*). Moreover, the best treatment was given at a sonication length of 120 min with a sonication amplitude of 60%. The characteristics of the CRS liquid dye with the best treatment are presented in Table 7.

4. Conclusions

This study explored the potential of rice straw as a sustainable resource for black food dyes to overcome the challenges associated with traditional natural dye sources. Treatments with HCl, NaOH, and

pressurised NaOH were utilised to effectively reduce the silica content in carbonised rice straw (CRS), with the most significant reduction achieved using 25% NaOH under pressure, which brought the silica content down to 1.1%. This method also increased the carbon content, highlighting its dual effectiveness in purification and carbon enrichment. In addition, the effect of NaOH concentration on silica reduction was systematically investigated. The results showed that higher NaOH concentrations led to lower silica content at the expense of increasing sodium levels to 3.6%, necessitating further optimisation. To enhance dye stability, sonication proved effective in reducing particle size, as longer durations and higher amplitudes resulted in smaller particle sizes (D90 of 4.09 µm). Sonication decreased the sedimentation rate to 9%, indicating the improved stability of the suspension. The optimised sonication conditions yielded a liquid dye with desirable characteristics, including a uniform particle distribution and minimal sedimentation. The black food dye colour was #41403b (very dark greyish yellow) with an L* value of 26.8±0.02. The pH stability of the CRS dye further supports its potential for various food processing conditions.

Future research should focus on optimising the NaOH treatment process to fine-tune the concentration and process parameters to achieve an optimal balance between silica reduction and minimal sodium content such as post-treatment rinsing or ion-exchange, ensuring compliance with food safety standards. Expanding the application range of the CRS-based black food dye in various food products to evaluate its performance under different processing conditions and its acceptance in the food industry, perform cost-energy and regulatory pathway are also essential. Conducting long-term stability studies and comprehensive safety assessments will ensure the CRS-based dye's suitability for prolonged use in food applications. Particle size reduction of the CRS required further investigation to meet EFSA's standard.

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Authors' contributions

CRediT: **Erni Sofia Murtini**: Conceptualization, Data curation, Funding acquisition, Writing – original draft; **Hendrix Yulis Setyawan**: Formal analysis, Writing – original draft, Writing – review & editing; **Sudarminto Setyo Yuwono**: Formal analysis, Supervision, Validation; **Luluk Mamluhah**: Investigation, Methodology; **Lisa Fitri Rahayu**: Investigation, Visualization, Writing – original draft; **Mingming Zhu**: Formal analysis, Supervision, Writing – review & editing; **Chiemeka Onyeka Okoye**: Supervision, Writing – review & editing.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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About the authors

Dr. Erni Sofia Murtini is a senior lecturer at the Department of Food Technology, Universitas Brawijaya, Indonesia. She holds a Ph.D. degree from Oklahoma State University, Stillwater, USA, specializing in cereal science and traditional Indonesian food technology. Her research focuses on functional food innovation, natural colorants, and the processing of local agricultural commodities.

Dr. Hendrix Yulis Setyawan is a lecturer at the Department of Agroindustrial Technology, Universitas Brawijaya, with expertise in agroindustry-based energy conversion processes, particularly pyrolysis. He earned his Ph.D. from The

University of Western Australia and has conducted extensive research on renewable energy, bio-oil, and biochar derived from agricultural residues.

Prof. Sudarminto Setyo Yuwono is a professor in the Department of Food Technology, Universitas Brawijaya, specializing in food science, food engineering, and food processing. His expertise includes food analysis, processing technology, and engineering applications in food production.

Mrs. Luluk Mamluhah holds an undergraduate degree in chemistry and has expertise in food analysis. She has participated in various professional education and training programs related to laboratory analysis and quality assessment in food science. Her work supports integrated laboratory operations and applied food testing.

Ms. Lisa Fitri Rahayu is a lecturer in agricultural product processing and engineering, with an academic background from Universitas Brawijaya in Food Science and Technology (B.Sc.) and Agricultural Product Technology (M.Sc.). She is active in teaching and research in the areas of food processing and agro-based product development at Mulawarman University.

Dr. Mingming Zhu is a Reader in Chemical Engineering at Cranfield University, UK, and an Adjunct Research Fellow at The University of Western Australia. His research focuses on biomass conversion, low-carbon fuels, and advanced energy systems, with an extensive record of publications and industry-funded projects.

Dr. Chiemeka Onyeka Okoye is a Research Officer at the Centre for Energy, The University of Western Australia and holds a Ph.D. degree from the same university. He has over five years of interdisciplinary experience in teaching, research, process design, material characterization, and renewable energy development.

ORCID

Erni Sofia Murtini  <http://orcid.org/0000-0002-6221-7526>
 Hendrix Yulis Setyawan  <http://orcid.org/0000-0002-3513-977X>
 Sudarminto Setyo Yuwono  <http://orcid.org/0000-0003-3772-5972>
 Mingming Zhu  <http://orcid.org/0000-0002-3643-1799>
 Chiemeka Onyeka Okoye  <http://orcid.org/0000-0002-9568-9028>

Data availability statement

The data that support the findings of this study are available in cloud at <https://s.ub.ac.id/blackdyedata>. These data were derived from the following resources available in the public domain: [<https://s.ub.ac.id/blackdyedata>]. The authors confirm that the data supporting the findings of this study are available within the article or from the corresponding author, HYS, upon reasonable request.

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Murtini, Erni Sofia; Setyawan, Hendrix Yulis; Yuwono, Sudarminto Setyo; Mamluhah, Luluk; Rahayu, Lisa Fitri ...

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