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Extraction and Characterization of Cellulose from Arabica Spent Coffee Grounds

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ABSTRACT

Global coffee production generates significant amounts of spent coffee grounds (SCG), often discarded, creating environmental challenges. Cellulose, a major component of SCG, can be extracted for industrial applications, such as food packaging and bioplastics. This research aims to determine the optimal cellulose purification time for maximizing purity and characterize the physicochemical properties of the extracted cellulose for food industry use. The study investigates the effect of different purification times on cellulose purity. The highest purity (74.40%) was obtained at 180 minutes, with the extracted cellulose showing high crystallinity and favorable properties, such as low moisture content (3.49%), water absorption capacity (6.17%), and bulk density (0.2871 g/cm³). Characterization using SEM revealed the morphology of cellulose crystals, and analysis (Carr's index: 15.33%, Hausner ratio: 1.1811) confirmed the material's suitability for biocomposites in food applications. Color analysis (L*: 76.34, C*: 28.52, h*: 16.46, WI: 62.94) also highlighted the material's potential for use in food formulations. This research provides valuable data for further applications of cellulose derived from SCG.

Keywords: *Cellulose extraction; Characterization; Lignocellulose; Spent coffee grounds; Sustainability*

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ABSTRAK

Produksi kopi global menghasilkan jumlah ampas yang signifikan, yang sering kali dibuang dan menimbulkan masalah lingkungan. Selulosa, komponen utama ampas kopi, dapat diekstraksi dan digunakan untuk berbagai tujuan industri, termasuk kemasan makanan dan bioplastik. Penelitian ini berfokus pada waktu pemurnian yang berbeda terhadap kemurnian selulosa yang diekstraksi. Selulosa yang diekstraksi dikarakterisasi menggunakan scanning electron microscopy (SEM), water absorption capacity (WAC), pengukuran bulk density, tapped density, Hausner Ratio, Carr's Index, water solubility index (WSI), analisis komponen lignoselulosa metode Chesson-Datta, dan analisis warna. Penelitian ini menemukan bahwa waktu pemurnian lignin dan hemiselulosa memengaruhi hasil selulosa dengan kemurnian tertinggi (74,40%) diperoleh pada 180 menit. Selulosa yang diekstraksi menunjukkan kristalinitas tinggi dan sifat fisikokimia yang diinginkan sehingga cocok untuk aplikasi dalam industri pangan. Analisis SEM mengungkap morfologi serat selulosa, sementara WAC (6,17%), kadar air (3,49%), tapped density (0,3390 g/cm³), bulk density (0,2871 g/cm³), Hausner Ratio (1,1811), Carr's Index (15,33%), hasil analisis Chesson-Datta (hemiselulosa 1,26%, selulosa 74,4%, lignin 24,34%), serta hasil analisis warna (L*:76.34; C*:28.52; h*:16.46; WI:62.94) menunjukkan potensi material untuk digunakan dalam formulasi makanan.

Keywords: *Ampas kopi; Ekstraksi selulosa; Karakterisasi; Lignoselulosa; Sustainability*

INTRODUCTION

Coffee is one of the most popular beverages in the world, with an estimated production of around 105 billion tons per year. Indonesia is one of the largest coffee producers globally, ranking fourth [1]. According to previous research, 1 ton of coffee beans can produce 650 kg of spent coffee grounds (SCG) with limited use, resulting in a lot of food waste [2]. Food waste has a significant impact on the environment, economy, society, and food technology. Therefore, the development of food waste processing and management is needed [3], [4]. One of the components of SCG is cellulose, which is an abundant natural biopolymer. Cellulose can be isolated and extracted from coffee grounds using various methods, thus opening up new opportunities for its application in various industries, including the food industry [5].

Pure cellulose has various advantages in the food industry, such as the ability to improve product texture and stability, as well as environmentally friendly, biodegradable, and biocompatible properties [6]. Pure cellulose can also serve as a significant source of dietary fiber, offering health benefits such as enhancing digestive health and reducing the risk of chronic diseases, including diabetes and heart disease [5]. Therefore, producing pure cellulose from coffee grounds not only contributes to waste reduction but also to the development of valuable materials for the food industry.

Research on the extraction of cellulose from various types of waste has been using OPEFB (oil palm empty fruit bunch) [7], coffee husk, rice husk [8], and coffee grounds [5], [9], where the purification time of cellulose was not a research variable. In addition, these studies employed acid hydrolysis methods, which are less environmentally friendly than using strong bases because they generate additional waste during the extraction process.

Extracting cellulose from coffee grounds involves multiple stages, including grinding, drying, delignification, bleaching, and purification. However, various factors can lower cellulose purity [2]. Thus, optimizing the extraction process is essential to obtain high-purity cellulose and to evaluate the resulting bio-composites for food applications.

LITERATURE REVIEW

Spent Coffee Grounds Potential

Coffee is one of the most popular beverages globally, with an estimated annual production of around 105 billion tons. Indonesia ranks as the fourth-largest coffee producer in the world [1]. One ton of coffee beans can produce up to 650 kg of spent coffee grounds (SCG), which are often discarded by burning or landfilling and pose environmental challenges due to their limited application which shows the urgency of repurposing these SCG into renewable material or alternative energy to reduce the environmental impact that it poses in indonesia [2]. One of the main components of SCG is cellulose, an abundant natural biopolymer that can be isolated and extracted using various methods, opening up new opportunities for its application in various industries, including the food industry [5].

Chemical Composition of Spent Coffee Grounds

Spent coffee grounds contain various organic compounds such as lipids, carbohydrates, carbon, nitrogen, and polyphenols [10]. The chemical composition of SCG includes protein, lipids, minerals, carbohydrates, caffeine, cellulose, hemicellulose, and lignin [11]. The presence of lignin can hinder the access of chemical reagents to cellulose, complicating the extraction process [2]. Hemicellulose, consisting of shorter and more branched sugar chains than cellulose, can also form hydrogen bonds with cellulose, making separation more difficult [12]. Pectin, which has gelling properties and can form a gel in acidic conditions, can also interfere with the extraction process due to its viscosity [13]. Additionally, proteins and polyphenols in SCG can form complexes with cellulose, further complicating the extraction process [10]. Cellulose is a significant polymer in the food industry, particularly in food packaging. It is found in plant cell walls and other organs such as fruits, vegetables, and leaves, playing a crucial role in plant cell structure [14]. Cellulose is a linear homopolymer composed of d-glucopyranose units connected by β-(1–4) glycosidic bonds, with numerous hydrogen bonds providing mechanical strength and making cellulose semi-rigid [15]. According to Franca & Oliveira, SCG contains 16–25% cellulose, indicating its potential for high-value applications [11].

Extraction Methods of Cellulose

The chemical extraction methods using strong bases involve several stages, including preparation, delignification, bleaching, and purification were used in this work. Sample preparation typically consists in drying and grinding the SCG. Delignification aims to remove lignin from SCG using chemical reagents like NaOH or KOH, opening up the lignocellulosic structure and allowing chemical reagents to access cellulose. NaOH can break hydrogen bonds between lignocellulosic components, causing lignin, hemicellulose, and lower molecular fractions to dissolve in the alkaline solution as black liquor, thereby increasing the yield of cellulose fibers.

Bleaching is the process of whitening cellulose using oxidative reagents such as $NaClO_2$ or H_2O_2 to remove or lift residual lignin after delignification. $NaClO_2$ and H_2O_2 consist of hypochlorite ions, which are strong oxidants capable of breaking ether bonds in lignin structure, thereby increasing overall brightness, as shown in Equation 1.

$$Lignocellulose + H_2O_2(s) \rightarrow Cellulose_{(s)} + Soluble\ oxidized + H_2O \tag{1}$$

Purification is carried out using NaOH solution and acetic acid, aiming to remove hemicellulose and increase the purity of extracted cellulose [16]. Cellulose does not dissolve when the extraction temperature is lower than its degradation temperature due to strong intramolecular and intermolecular hydrogen bonds in its structure. Therefore, in the purification process, temperature treatment is applied to accelerate the reaction [17]. The reaction between strong bases and SCG can be seen in Equation 2.

$$SCG + NaOH_{(aq)} \longrightarrow Cellulose_{(s)} + Hemicellulose\ sugars + Lignin - Na\ salts \tag{2}$$

In the use of strong acids such as H_2SO_4, hemicellulose is hydrolyzed into oligomeric and monomeric sugars. Strong acids can dissolve lignin by breaking down its complex structure, in which the aromatic rings and side chains of lignin undergo ring-opening reactions and cleavage of C–O–C bonds, resulting in smaller, more soluble fragments. Although strong acids are effective in disrupting the lignocellulosic structure, this approach presents several drawbacks, such as being environmentally unfriendly due to the corrosive nature of the acids, making it less suitable for industrial applications [18]. Additionally, reaction by-products such as furfural and hydroxymethylfurfural (HMF) may inhibit microbial fermentation processes, which can hinder enzymatic hydrolysis when a combined extraction method is applied. Enzymatic routes can also be employed for cellulose extraction. In an enzymatic extraction process, specific enzymes selectively depolymerise non-cellulosic constituents, mainly lignin and hemicellulose so that cellulose can be recovered more efficiently and with a smaller environmental footprint, as the method avoids harsh chemicals. The enzymes most frequently applied in enzymatic cellulose extraction are cellulases, hemicellulases, and ligninases, each playing a distinct role in disassembling and removing non-cellulosic fractions from spent coffee grounds [14].

METHOD

Cellulose Extraction

Figure 1 shows the process diagram of cellulose extraction. SCG was collected within a day and stored at −8°C before use. The samples were oven-dried at 105°C for 30 minutes [16], cooled in a desiccator, ground, and weighed (30 g per treatment). Delignification was performed using 200 mL of 3% KOH at 90°C for 2 hours under mechanical stirring. The residue was washed and neutralized to pH 7. For bleaching, 200 mL of 10% H_2O_2 and 0.1 M NaOH were added until

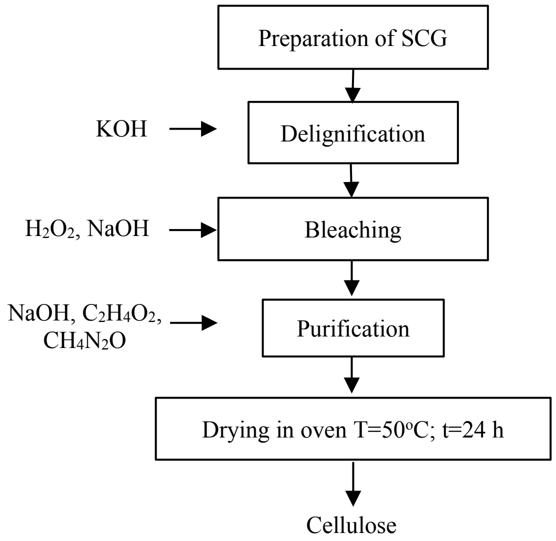


Figure 1. Flowchart of the cellulose extraction process.

the pH reached 9; the mixture was treated for 45 minutes, then washed to neutral pH. Further extraction was carried out with 8.5 g NaOH, 5 mL CH_3COOH, 8.5 g urea, and 100 mL distilled water at 70°C for 60, 120, and 180 minutes under continuous stirring. The resulting cellulose was filtered, washed, and dried at 50°C for 24 hours. Yield was calculated, and cellulose purity was assessed using the Chesson-Datta method.

Cellulose Yield & Purity

The Chesson-Datta method [19] was used to determine the physicochemical composition of extracted cellulose from SCG, including cellulose, hemicellulose, and lignin. Three-step purification was performed: (a) hot water treatment, (b) 1N H_2SO_4 hydrolysis, and (c) 72% H_2SO_4 delignification, followed by washing, drying, and gravimetric analysis at three times intervals (60, 120, and 180 min) to identify the highest cellulose yield. The best treatment was selected based on the highest cellulose purity (%) derived from Chesson-Datta data across three purification times.

Microscopic Characterization

SEM (Scanning Electron Microscopy) was employed to observe the surface morphology and crystalline structure of the extracted cellulose, providing detailed insights into its microstructural characteristics. The analysis was performed using an electron beam at a voltage of up to 30 kV [20], allowing for high-resolution imaging of the cellulose samples.

Compressibility and Flowability Characteristics

Bulk density was determined by measuring the volume of 1 g of cellulose powder in a graduated cylinder [21]. Tapped density was measured after 50 taps on the cylinder containing 1 g sample to assess the compressibility of the [21]. Hausner ratio was obtained from the ratio of tapped density to bulk density, which indicates powder flowability [21]. Carr's index evaluates compressibility and flow behavior under industrial conditions [21].

Physical and Color Analysis

WAC was measured by suspending a one-g sample in 10 g of water, incubating at room temperature for 30 min, and centrifuging at 6,000 rpm for 15 min [22]. Moisture content was analyzed using a moisture analyzer on a one-g sample [23]. WSI was evaluated by centrifuging a one g sample in 10 g of water at 6000 rpm for 15 min, drying the supernatant at 105°C, and calculating solubility [24]. Color parameters (Chroma (C*), Hue Angle (h*), and Whiteness Index (WI) were determined using a colorimeter [25].

Experiment Design

Table 1. Experimental design.

Treatment	Repetition		
	U1	U2	U3
P1	P1U1	P1U2	P1U3
P2	P2U1	P2U2	P2U3
P3	P3U1	P3U2	P3U3

Note: P1=60 minutes purification time; P2=120 minutes purification time; P3=180 minutes purification time.

Chesson-Datta analysis and selection of the optimal treatment were conducted using one-way ANOVA ($\alpha = 0.05$), followed by Tukey's HSD post-hoc test for significant differences. An independent samples t-test was applied for color analysis. The best treatment was determined based on significant differences from the control and the highest cellulose purity. Statistical analyses were performed using IBM SPSS Statistics 26.

RESULTS AND DISCUSSION

Effect of Purification Time on Cellulose Yield and Purity

Table 2. Extraction and Chesson-Datta analysis results.

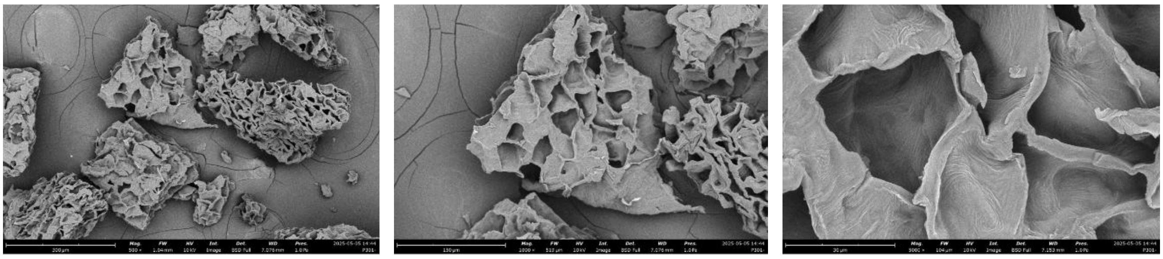
Variable	Yield (%)	Hemicellulose (%)	Cellulose (%)	Lignin & others (%)
SCG	-	5.00 ± 0.25^{c}	30.30 ± 0.04^{a}	64.69 ± 0.24^{a}
P1	24.90^{b}	2.61 ± 0.75^{ab}	61.85 ± 0.36^{b}	35.54 ± 0.52^{b}
P2	20.42^{a}	3.19 ± 0.98^{b}	67.28 ± 0.48^{c}	29.53 ± 0.98^{c}
P3	19.53^{a}	1.26 ± 0.05^{a}	74.40 ± 0.45^{d}	24.34 ± 0.42^{d}

Note: SCG=Spent Coffee Grounds; P1=60 minutes purification time; P2=120 minutes purification time; P3=180 minutes purification time. The percentage of hemicellulose, cellulose, and lignin is based on a 1-gram sample. The data are presented as mean ± standard deviation. Identical notation in each column indicates no significant differences among the treatments at $p \geq 0.05$.

The study investigated the impact of different purification times on the yield and purity of cellulose extracted from spent coffee grounds (SCG). The results showed that the yield and purity of cellulose were significantly influenced by the purification time. If the purification time for hemicellulose is too short, cellulose may remain bound to hemicellulose and lignin; if too long, cellulose degradation may occur, both reducing purity. Additionally, improper chemical reagents or suboptimal extraction conditions can lead to impure yields [2]. A longer purification time can enhance cellulose purity due to the amphiphilic nature of cellulose, which influences its interactions and leads to the formation of highly hydrated and coordinated ionic structures that disrupt hydrogen bonding [16]. As a result, extended purification may degrade the components that bind cellulose, thereby reducing the percentage of lignin and hemicellulose and increasing the purity of the extracted cellulose. The highest cellulose purity of 74.40% was achieved at a purification time of 180 minutes but with a low yield of 19.53%, whereas the highest yield was achieved at a purification time of 60 minutes, but it contains a significantly lower cellulose purity value at 61.85%. This indicates that longer purification times are more effective in removing non-cellulosic components such as lignin and hemicellulose, thereby increasing the purity and lowering the yield of the extracted cellulose [1].

Microscopic Characterization

Scanning electron microscopy (SEM) was used to observe the morphology of the extracted cellulose. The SEM analysis revealed that the cellulose crystals had a regular and smooth structure, indicating high crystallinity (Figure 2). The presence of porous areas and rough surfaces suggested a high surface area, which is beneficial for applications requiring water absorption or binding with other compounds. The crystal size ranged from 80 to 300 µm, indicating that the inter-particle spaces were relatively small, contributing to the material's compressibility and flowability [20].



(a) (b) (c)

Figure 2. SEM image of cellulose: (a) 500× magnification, (b) 1,000× magnification, and (c) 5,000× magnification.

Compressibility and Flowability Characteristics

The bulk density and tapped density of the extracted cellulose were measured to evaluate the material's packing efficiency and flow properties. The bulk density was found to be 0.2792 g/cm³, which is considered low (<0.3 g/cm³) (Table 3). This low bulk density indicates that the cellulose has a loose structure, making it easier to process in applications requiring good flow properties. The tapped density was 0.3301 g/cm³, which is higher than the bulk density, indicating that the cellulose can be easily compressed into a denser form. This property is advantageous for applications such as packaging, where compressibility is important. The Hausner ratio and Carr's index were calculated to assess the flowability and compressibility of the cellulose powder. The Hausner ratio was 1.1827, which is close to 1, indicating good flow properties. The Carr's index was 15.44%, which is below 20%, indicating good compressibility. These results suggest that the extracted cellulose has desirable flow and compressibility characteristics for industrial applications [21].

Table 3. Bulk density, Tapped density, Hausner Ratio, and Carr's Index results.

Sample	Bulk Density (g/cm^3)	Tapped Density (g/cm^3)	Hausner Ratio	Carr's Index
P3	0.2792 ± 0.02	0.3301 ± 0.02	1.1827 ± 0.01	15.44 ± 0.53

Note: P3=180 minutes purification time. The data are presented as mean ± standard deviation.

Physical Properties

The water absorption capacity (WAC) of the extracted cellulose was measured to determine its ability to absorb water. Table 4 shows that the WAC was found to be 6.15%, which is above the threshold of 5%. This high WAC indicates that the cellulose has excellent water absorption properties, making it suitable for applications that require moisture retention, such as in food products. The water solubility index (WSI) of the extracted cellulose was measured to assess its solubility in water. The WSI was found to be 15.09%, which is below the threshold of 20%. This low WSI indicates that cellulose has limited solubility in water, making it suitable for applications that require water resistance, such as in packaging materials [6]. The moisture content of the extracted cellulose was determined to be 3.47%, which is below the threshold of 5%. This low moisture content indicates that the cellulose is stable in humid environments and has good mechanical properties. However, it may require additional processing to improve its flexibility for certain applications [26].

Table 4. Water absorption capacity, water solubility index, and moisture content results.

Sample	WAC (%)	WSI (%)	Moisture Content (%)
P3	6,15 ± 0,13	15,09 ± 0,63	3,47 ± 0,04

Note: P3=180 minutes purification time. The data are presented as mean ± standard deviation.

The water absorption capacity (WAC) of the extracted cellulose was measured to determine its ability to absorb water. The WAC was found to be 6.15%, which is above the threshold of 5%. This high WAC indicates that the cellulose has good water absorption properties, making it suitable for applications requiring moisture retention, such as in food products. The water solubility index (WSI) of the extracted cellulose was measured to assess its solubility in water. The WSI was found to be 15.09%, which is below the threshold of 20%. This low WSI indicates that the cellulose has limited solubility in water, making it suitable for applications requiring water resistance, such as in packaging materials [6]. The moisture content of the extracted cellulose was determined to be 3.47%, which is below the threshold of 5%. This low moisture content indicates that the cellulose is stable in humid environments and has good mechanical properties. However, it may require additional processing to improve its flexibility for certain applications [26].

Color Analysis

Table 5 shows the color analysis of SCG and cellulose. SCG has a very dark color with an L* value of 19.21 and a WI of 16.45, indicating that the coffee grounds are not very white. The Chroma value of 20.56 indicates relatively low color strength, and the Hue angle of 35.57° means that the color of the coffee grounds tends towards orange. This color analysis suggests that the cellulose is still bound with other components, such as lignin and hemicellulose, which give the sample a dark color. In contrast, the P3 sample has a much brighter color compared to SCG, with an L* value of 76.34 and a WI of 62.94. The high L* value indicates that the extracted cellulose is very bright, and the high WI value indicates that the extracted cellulose is quite white. The Chroma value of the extracted cellulose is 28.52, indicating a higher color strength. The Hue angle of 16.46° reveals that the color of the cellulose tends towards yellow. This color analysis proves that the extraction process has successfully removed most of the lignin and hemicellulose. The delignification process with KOH breaks down lignin bonds, and bleaching with H_2O_2 oxidizes the remaining lignin, thereby increasing the brightness of the extracted cellulose [16].

Table 5. Color analysis results.

Sample	L*	Chroma (C*)	Hue Angle (h*)	Whiteness Index (WI)	Color Description	
					Name	Hex code
SCG	19.21 ± 0.52	20.56 ± 0.82	35.57 ± 1.44	16.45 ± 0.56	bistre	#452716
P3	76.34 ± 0.64	28.52 ± 0.77	16.46 ± 0.24	62.94 ± 0.96	burlywood	#DCB58A

Note: SCG=Spent Coffee Grounds; P3=180 minutes purification time. The data are presented as mean ± standard deviation.

In a study conducted by Sari et al., it was found that cellulose extracted with H_2O_2 had a higher brightness level compared to the cellulose from coffee grounds obtained [27]. This could be due to differences in the concentration of bleaching agents used, where OPEFB cellulose was extracted using a higher concentration compared to coffee grounds cellulose. Additionally, the material used also affects the final brightness obtained. Commercial cellulose has a very high brightness (L* = 100) and a whiter and more neutral color because it has undergone a very high purification process, resulting in cellulose with optimal purity and brightness. This indicates that brightness is directly proportional to cellulose purity, where the higher the brightness value, the higher the purity of the cellulose obtained. In the context of the food industry, this color property is very important as it can affect consumer preferences and the quality of the final product. Bright and white cellulose may be more desirable for applications such as thickeners and binders in food products, raw materials for bioplastics, or bio-composites. However, it may require additional processing to improve its flexibility for certain applications.

CONCLUSION

The study concludes that spent coffee grounds can be a valuable source of cellulose for food industry applications. The optimal purification time of 180 minutes yields high-purity cellulose with desirable physicochemical properties. The extracted cellulose exhibits high crystallinity, good flowability, compressibility, water absorption capacity, and limited water solubility, making it suitable for various food industry applications, especially in food packaging for the development of bioplastics. Further research is recommended to explore the application of this cellulose in specific food products and to develop more environmentally friendly extraction methods.

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