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Optimization of holocellulose extraction from spent coffee grounds using factorial design

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Abstract. Spent coffee grounds (SCG) represent a substantial environmental challenge due to the massive generation of solid waste and its high organic load. However, SCG is a promising source of lignocellulosic biomass for the production of high-value biopolymers. This study aimed to optimize the extraction of holocellulose from SCG via oxidative bleaching using sodium hypochlorite (NaOCl), employing a 2^3 factorial design with a central point. The effects of NaOCl concentration (1.0–2.5%), reaction time (15–45 min), and temperature (25–55 °C) on the extraction yield were evaluated. Statistical analysis revealed that temperature was the most significant variable, accounting for approximately 52% of the yield variation, followed by NaOCl concentration (∼37%). High temperatures and concentrations led to excessive oxidative degradation and hydrolysis of the polysaccharide chains, drastically reducing yields to below 20%. Conversely, mild conditions resulted in insufficient delignification. The experimental results indicated that the central point conditions (1.75% NaOCl at 40 °C for 30 min) offered the optimal balance, providing a yield of approximately 38% with adequate brightness and reduced degradation. These findings demonstrate the feasibility of using statistical design to obtain holocellulose from SCG for future applications in biocomposites and packaging.
Keywords: sodium hypochlorite, factorial design, oxidative bleaching.

Optimizarea extracției holocelulozei din zaț de cafea prin plan factorial

Rezumat. Zațul de cafea (SCG) reprezintă o provocare semnificativă pentru mediu datorită generării masive de deșeuri solide și a încărcăturii organice ridicate a acestora. Totuși, SCG este o sursă promițătoare de biomasă lignocelulozică pentru producerea de biopolimeri cu valoare ridicată. Acest studiu a vizat optimizarea extracției holocelulozei din SCG prin albire oxidativă utilizând hipoclorit de sodiu (NaOCl), folosind un plan factorial 2^3 cu un punct central. Au fost evaluate efectele concentrației de NaOCl (1.0–2.5%), timpului de reacție (15–45 min) și temperaturii (25–55 °C) asupra randamentului extracției. Analiza statistică a relevat că temperatura a fost cea mai semnificativă variabilă, fiind responsabilă pentru aproximativ 52% din variația randamentului, urmată de concentrația de NaOCl (∼37%). Temperaturile și concentrațiile ridicate au dus la o degradare oxidativă excesivă și la hidroliza lanțurilor polizaharidice, reducând drastic randamentele sub 20%. În schimb, condițiile blânde au rezultat într-o deslignificare insuficientă. Rezultatele experimentale au indicat că condițiile punctului central (1.75% NaOCl la 40 °C timp de 30 min) au oferit echilibrul optim, asigurând un randament de aproximativ 38% cu un grad de alb adecvat și o degradare redusă. Aceste constatări demonstrează fezabilitatea utilizării planificării statistice pentru obținerea holocelulozei din SCG pentru aplicații viitoare în biocompozite și ambalaje.

Cuvinte-cheie: hipoclorit de sodiu, plan factorial, albire oxidativă.

1. Introduction

Coffee is one of the most widely consumed beverages worldwide, being commercialized in over 80 countries. However, spent coffee grounds (SCG), the solid residue obtained after brewing, stand out as one of the major waste products from coffee beverage preparation, representing an estimated 60 million tons of discarded grounds globally. Thus, the improper disposal of SCG constitutes a significant environmental issue, given that it is often disposed of in landfills. This practice has a high pollution potential due to the residue's excessive organic matter load, which consequently demands a large amount of oxygen for its decomposition [1], [2].

SCG is a lignocellulosic material rich in lignin, hemicellulose, cellulose, proteins, and lipids, making it a potential raw material for applications in various sectors. Among these polysaccharides, holocellulose (composed of hemicellulose and cellulose) shows potential for use in biocomposites and the packaging industry due to its stable thermal and mechanical properties. However, the removal of lignin from the material's structure is a necessary prerequisite [3].

Sodium hypochlorite (NaOCl) is a well-known oxidizing agent used for removing lignin from biomass to obtain holocellulose, which consists of hemicellulose and cellulose. However, severe conditions can lead to biomass hydrolysis, reducing yield and compromising the integrity of the polymeric chains [4].

Therefore, this work aims to optimize the extraction of holocellulose from SCG via oxidative bleaching by varying NaOCl concentration, time, and temperature. The goal is to identify, using statistical analysis, the optimal delignification condition without causing excessive polysaccharide degradation.

2. Materials and methods

SCG were supplied by the company *Café Brasileiro* in powder form. The material was defatted using a hexane/ethanol mixture (50:50 v/v) for 48 hours to remove extractives, such as oil, followed by drying in an oven at 105 °C for 12 hours. For the bleaching of the defatted SCG, commercial bleach (*Candura* brand) containing 2.5% NaOCl was used. Experiments were conducted using 0.4 g of defatted SCG and 50 mL of NaOCl solution at the desired concentration, under agitation, for specific times and temperatures as detailed in Table 1. After the reaction, samples were filtered, washed until a neutral pH was reached, and dried in an oven at 70 °C for 12 hours. To determine bleaching efficiency, the yield was calculated based on the initial and final mass of each sample.

Statistical analysis was performed using the open-source software Octave to determine the analysis of variance, experimental error, effect error, and the percentage contribution of effects, as described by [5]. Response surface and contour plots were generated from the model coefficients, calculated as half the value of each observed effect, while the coefficient b_0 represents the mean of the 8 experiments. The plots were generated using the command `[superficie]=super_fabi2(X,passo)`, where *X* refers to the input data matrix and 'step' corresponds to the numerical refinement (resolution) of the generated surface.

3. Results and discussion

Figure 1 shows the samples obtained after the bleaching of spent coffee grounds under the different conditions evaluated, with varying NaOCl concentrations, times (min), and temperatures (°C). Depending on the experimental conditions tested, the samples ranged in color from dark to light, indicating the efficiency of the delignification process using commercial 2.5% NaOCl.

Figure 1. Samples obtained after bleaching under all conditions

Table 1 presents the yields obtained for the 2^3 factorial design with all possible combinations, plus the central point for determining the effect error and data reliability. The results indicate that low NaOCl concentrations and short reaction times result in higher yields but indicate low delignification efficiency, as the samples remain slightly brown. Conversely, high NaOCl concentrations combined with high temperatures promote excessive delignification and potential hydrolysis of cellulose chains, resulting in extremely low holocellulose yields (ranging from 13% to 20%). As reported by [6] at high temperatures during NaOCl treatment, competitive oxidation between lignin and cellulose occurs, favoring the oxidative degradation of holocellulose, which results in significantly lower yields.

Table 1. Yield obtained from the 2^3 factorial design plus central point

Experiment	NaOCl (%)	Time (min)	Temperature (°C)	Yield (%)
1	1	15	25	57.13
2	2.5	15	25	45.73
3	1	45	25	51.19
4	2.5	45	25	44.55
5	1	15	55	42.09
6	2.5	15	55	21.17
7	1	45	55	41.17
8	2.5	45	55	13.19
9	1.75	30	40	37.25
10	1.75	30	40	37.79
11	1.75	30	40	38.99

As seen in Figure 2 (A), which presents the percentage contribution of the 7 possible effects for a 2^3 factorial design (covering primary effects and secondary/tertiary interactions), the predominant effect on holocellulose yield is effect 3, related to temperature, accounting for approximately 52% of the observed variation. The second most relevant effect is associated with NaOCl concentration, contributing approximately 37%. Thus, the effects of temperature and NaOCl concentration together account for nearly 90% of the yield variation for holocellulose obtained from SCG. These results differ from those reported for NaOCl treatments of eucalyptus wood, where authors found that temperatures between 30 and 50 °C did not have a significant effect on substrate yield [6]. Conversely, effect 2, referring to time, did not demonstrate a high impact, accounting for only 2–3% of the analyzed response. Figure 2 (B) presents the probability plot and the magnitude

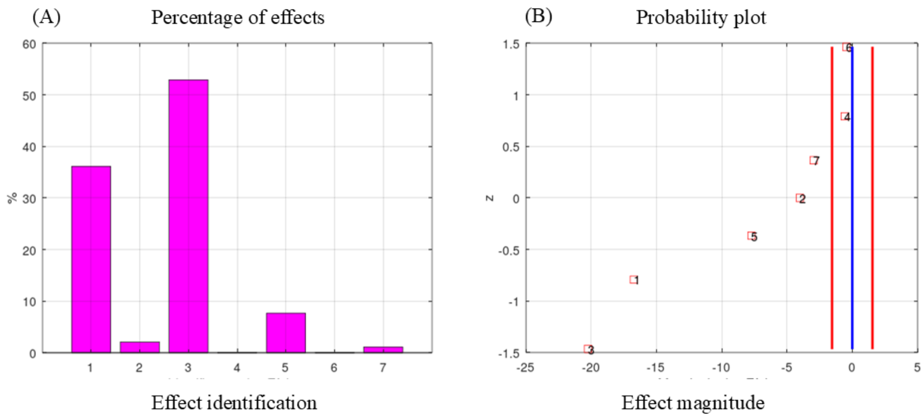

Figure 2. Effects percentage plot (A) and probability plot (B)

of each effect on sample yield. It is notably observed that effect 3, related to temperature, plays a significant role in the yield: employing higher temperatures (e.g., 55 °C) can reduce the obtained yield by up to 20%. Subsequently, effect 1, regarding NaOCl concentration, is also highly significant; high concentrations (e.g., 2.5%) lead to a yield reduction of approximately 17%. High NaOCl concentration combined with elevated temperature can promote intense hydrolysis of cellulose chains, not only removing lignin but also degrading the main cellulose structure, as evidenced by the low yield values shown in Table 1 [6], [7].

Effect 2 (time), despite its low percentage contribution shown in Figure 2 (A), proved to be statistically significant regarding the observed yield. Thus, prolonged times for holocellulose extraction, such as 55 minutes, can reduce the material yield by up to 4%. Furthermore, in the probability plot (Figure 2 B), the error interval is defined by the red vertical lines surrounding the blue line. All effects falling within this interval are not statistically significant; in this case, these are effects 4 and 6, corresponding to the secondary interactions of concentration/time and time/temperature, respectively [5].

Figure 3 (A) presents the response surface plot featuring the two most significant variables for yield, while Figure 3 (B) shows the corresponding contour plot with a color bar.

By analyzing both figures, it is observed that obtaining higher holocellulose yields requires low temperatures (e.g., 25 °C) and low NaOCl concentrations (e.g., 1%). However, despite yields exceeding 50% under these conditions, it is noteworthy that delignification may be insufficient, resulting in a material that retains residual lignin.

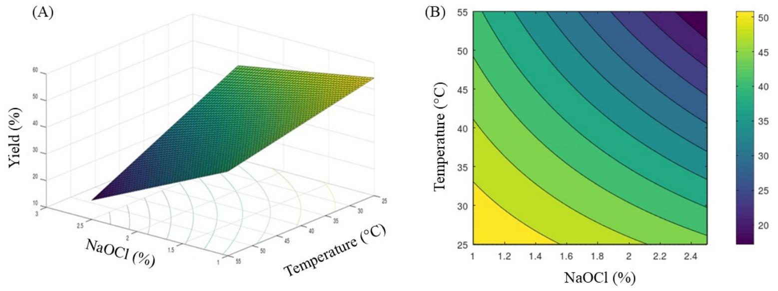

Figure 3. Response surface plot (A) and contour plot (B)

At the opposite extreme, a NaOCl concentration of 2.5% and a temperature of 55 °C result in a yield of approximately 20%, indicating intense oxidative degradation and scission of cellulose and hemicellulose chains [8]. Therefore, to balance yield and effective delignification without excessive degradation, the optimal point lies at the experimental center point (1.75% NaOCl and 40 °C). This condition provided a yield of approximately 40%, as shown in Table 1, in addition to higher brightness.

4. Conclusions

The present study successfully optimized the holocellulose extraction process from spent coffee grounds utilizing a factorial design approach. The statistical analysis demonstrated that temperature and NaOCl concentration are the determining factors in the bleaching process, together accounting for nearly 90% of the observed yield variation, whereas reaction time showed a minor impact. It was concluded that while aggressive conditions (55 °C, 2.5% NaOCl) maximize delignification, they concurrently promote severe hydrolysis and scission of cellulose and hemicellulose chains, rendering the process inefficient due to low yields. On the other hand, the central point conditions (1.75% NaOCl, 40 °C, 30 min) provided the most effective balance between yield retention and lignin removal. Therefore, this optimized method allows for the recovery of high-quality holocellulose from agri-food waste, offering a sustainable alternative for the development of green materials and reducing the environmental impact of coffee industry residues.

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