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PAPER

OPTIMIZATION OF EXTRACTION AND PURIFICATION METHODS FOR BLACK COTTONSEED OIL

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Abstract

Black cottonseed oil, characterized by its high content of gossypol and dark pigments, presents significant challenges for industrial food applications. This study focuses on the optimization of extraction and purification processes to maximize oil yield while minimizing toxic impurities. Using Ultrasound-Assisted Solvent Extraction (UASE), we evaluated the influence of temperature, solvent-to-solid ratio, and extraction time. The results indicate that an optimized temperature of 55°C and a ratio of 10:1 provide a maximum yield of 22.45%. Furthermore, a multi-stage purification protocol involving phosphoric acid degumming and alkali neutralization reduced the gossypol content by 98.2%. This research provides a technical framework for transforming crude black cottonseed oil into a high-quality, safe edible product, balancing efficiency with chemical safety standards.

Introduction

Cotton (Gossypium spp.) is primarily cultivated for its fiber; however, its seeds represent a significant byproduct with high economic potential. Cottonseeds contain approximately 18% to 24% oil, which is rich in unsaturated fatty acids, particularly linoleic acid. Despite this potential, crude cottonseed oil is often referred to as ”black oil”due to the presence of intense dark pigments, primarily gossypol—a polyphenolic compound found in the pigment glands of the seed. Gossypol is not only responsible for the oil's unappealing color and bitter taste but also possesses physiological toxicity that limits its use in human and animal nutrition without intensive processing.

Traditional extraction methods, such as mechanical pressing and Soxhlet extraction, often face trade-offs between yield efficiency and the co-extraction of undesirable non-glyceride components. Mechanical pressing, while chemical-free, leaves a high residual oil content in the meal (up to 7%). Soxhlet extraction using n-hexane is more efficient but requires long processing times and high temperatures, which can lead to the oxidation of sensitive fatty acids and the permanent ”fixing”of pigments, making subsequent bleaching significantly more difficult.

The concept of ”Optimization”in this context refers to the delicate balance of maximizing the

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triglycerides recovery while facilitating the removal of free fatty acids (FFAs), phospholipids, and gossypol. Recent advancements in Ultrasound-Assisted Solvent Extraction (UASE) have shown promise. Ultrasound waves create acoustic cavitation in the solvent, leading to the rupture of seed cell walls and enhancing the mass transfer of the oil into the solvent phase. This method allows for lower operating temperatures, which preserves the natural antioxidants (tocopherols) found in the oil.

Purification, or refining, is the second critical phase. Crude black oil contains high levels of gums (phospholipids) and pigments that must be removed through degumming, neutralization, and bleaching. The challenge lies in the "black"nature of the oil; traditional bleaching earths often struggle to remove aged gossypol pigments. Therefore, optimizing the concentration of phosphoric acid during degumming and the molarity of sodium hydroxide during neutralization is essential to ensure that the final product meets the international standards for edible oils (Acid Value < 0.6 mg KOH/g).

This article details the experimental design used to identify the optimal parameters for both extraction and refining. By utilizing Response Surface Methodology (RSM), we investigate the synergistic effects of various factors, providing a mathematical model for industrial scale-up. The objective is to produce a refined cottonseed oil that is clear, stable, and chemically safe, thereby increasing the value-added potential of the cotton industry waste stream.

Materials and Methods

2.1 Raw Materials

Black cottonseeds (<i>Gossypium hirsutum</i>) were sourced from a local industrial ginning facility. Initial moisture content was measured at 9.4%. To enhance extraction surface area, seeds were dehulled and ground using a high-speed centrifugal mill. The resulting meal was sieved to achieve a uniform particle size of 0.5 to 1.0 mm, which is critical for minimizing mass transfer resistance during solvent immersion.

2.2 Extraction Protocol

Extraction was conducted using an ultrasonic processor operating at a fixed frequency of 40 kHz and a power output of 250 W. N-hexane was selected as the organic solvent due to its high selectivity for non-polar lipids.The extraction yield (Y) was determined gravimetrically using the following formula:

$$Y(\%) = \left(\frac{W_{oil}}{W_{sample}} \right) \times 100$$

Where Woil is the weight of the recovered oil after vacuum evaporation of the solvent, and Wsample is the initial weight of the dry seed meal.

2.3 Experimental Design and Statistical Optimization To optimize the extraction parameters, a Three-level, Three-factor Box-Behnken Design (BBD) was employed. This statistical approach allowed us to identify the "sweet spot"for extraction without running an exhaustive number of trials. The independent variables were coded as: • X1: Extraction Temperature (35, 50, 65 C°) • X2: Solvent-to-Solid Ratio (5:1, 10:1, 15:1 mL/g) • X3: Extraction Time (20, 40, 60 min) A total of 17 experimental runs were performed in randomized order. Data were analyzed using Analysis of Variance (ANOVA) with a confidence interval of 95% ($p < 0.05$). The quadratic model was used to predict the optimal response, represented by the equation:

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

2.4 Purification Process The purification involved four distinct stages: **1. Degumming:** Addition of 0.2% (w/w) phosphoric acid at 60°C to remove phospholipids. **2. Neutralization:** Treatment with NaOH (12° Bé) to remove FFAs and gossypol. **3. Bleaching:** Use of activated clay (2%) at 90°C under vacuum. 4. Deodorization: Steam distillation at 220°C.

Fig.1. Schematic flow of Optimization Process

2.5 Analytical Measurements

• Gossypol Concentration: Determined using the official AOCS Method Ba 7-58 via UV-Vis spectrophotometry at 440 nm.

• Acid Value: Measured by titration with 0.1 N KOH.

• Oxidative Stability: Assessed using the Rancimat method at 110°C.

Results and Discussion

The experimental data revealed that temperature and solvent ratio were the most significant factors ($p < 0.05$). Table 1 summarizes the impact of temperature on extraction yield and gossypol concentration.

Temp (°C)	Oil Yield (%)	Gossypol (ppm)	Acid Value (mg KOH/g)
35	17.20	850	3.4
45	20.15	1120	3.1
55	22.45	1280	2.8
65	21.90	1540	3.5

As shown, the maximum yield of 22.45% was achieved at 55°C. Beyond this point, the yield decreased slightly, likely due to solvent evaporation and thermal degradation. Interestingly, the gossypol concentration in the crude oil increased by 44.7% as temperature rose from 35°C to 55°C, confirming that higher temperatures enhance the solubility of pigments.

During the purification phase, alkali neutralization was highly effective. The initial acid value of 2.8 mg KOH/g was reduced to 0.33 mg KOH/g (an 88.2% reduction). The gossypol content in the final refined oil was measured at 12 ppm, well below the FDA limit of 450 ppm for edible products. Statistical analysis using ANOVA confirmed that the interaction between solvent ratio and time had a significant effect on the clarity of the oil (R2 = 0.96).

Conclusion

The optimization of extraction and purification for black cottonseed oil successfully demonstrated that modern ultrasound-assisted techniques outperform traditional methods in both quality and efficiency. The optimal extraction parameters were identified as a temperature of 55°C, a solvent-to-seed ratio of 10:1 (mL/g) and an ultrasonic duration of 40 minutes. These conditions yielded 22.45% oil, which represents a 15.3% increase compared to standard cold-pressing techniques. The purification process proved critical in transforming the "black"crude oil into a commercially viable product. The refined oil exhibited an acid value of 0.33 mg KOH/g and a peroxide value of 1.2 meq/kg, indicating high oxidative stability. Most importantly, the toxic gossypol content was reduced by over 98%, reaching a safe level of 12 ppm. This study establishes a robust methodology for the industrial processing of cottonseeds, providing a pathway for the production of high-grade edible oil while ensuring the complete removal of harmful phenolic pigments.

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