

Research Article

# Pilot Study to Assess the Contamination of Cottonseed Oil Produced in Mali by Organochlorine Pesticide Residues

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## Abstract

In Mali, cotton cultivation generates large quantities of cottonseed, from which edible cottonseed oil is widely produced and consumed. As part of national food safety surveillance, sanitary standards and market authorization requirements are being strengthened, necessitating routine pesticide residue assessments. This study aimed to evaluate the presence and levels of organochlorine pesticide residues in cottonseed oil marketed in Mali. A total of 27 cottonseed oil samples from different processing units were collected and anonymized by coded labeling. Laboratory analyses were conducted at the Environmental Toxicology and Quality Control Laboratory (ETQCL). Pesticide residues were extracted using the laboratory's standard liquid–liquid extraction protocol, and quantification was performed using gas chromatography equipped with an electron capture detector (GC– $\mu$ ECD). Given the persistence and bioaccumulation potential of organochlorines, the investigation focused on this class of pesticides. Results indicated that 62.95% of samples were free of the targeted organochlorine residues, while 37.05% contained detectable levels. Eight out of the ten pesticides screened were identified, with concentrations ranging from 0.046 to 0.501 mg/L. DDT recorded the highest level (0.501 mg/L), whereas dieldrin showed the lowest (0.046 mg/L). Several detected concentrations exceeded the Codex Alimentarius maximum residue limits for edible oils. These findings demonstrate potential health risks associated with the consumption of contaminated cottonseed oil. It is therefore recommended to enhance pesticide regulation in cotton production, promote safer pest management alternatives, and establish regular monitoring systems to ensure oil safety for consumers.

## Keywords

Cottonseed Oil, Pesticides Residues, Organochlorine, Food Safety, Mali

## 1. Introduction

The introduction plays an important role in providing background information (including relevant references), emphasizing the importance of the study, and outlining its objectives.

It is crucial to conduct a thorough review of the current state of the research field and incorporate key publications into

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your work. By referencing other research papers, you can provide context and position your own work within the broader research landscape. The final paragraph should provide a concise summary of the main findings and conclusions, which will be helpful to the readers. Mali is a Sahelian country where agriculture represents the main driver of the national economy. It contributes approximately 35% to the Gross Domestic Product (GDP) and remains the primary source of income for more than 70% of the rural population [1]. Among cash crops, cotton holds a strategic position. Mali is one of the leading cotton producers in West Africa, with production reaching approximately 760,000 tons of seed cotton in the 2021–2022 season, placing the country among the top producers in Africa [2, 3].

In addition to fiber processing for export, cottonseed represents a valuable by-product, notably used in the production of edible oil. Cottonseed oil is widely consumed in Mali, where it is commonly used for cooking, seasoning, and frying [4]. Historically, cotton processing and oil production were dominated by the Compagnie Malienne de Développement des Textiles (CMDT) and the Huilerie Cotonniere du Mali (HUICOMA). These industries contributed significantly to the economic growth of cotton-producing regions for nearly two decades [5].

However, the cotton crisis of the early 2000s, followed by the privatization and decline of HUICOMA, resulted in the closure of several industrial units and severe socioeconomic impacts, including job losses [6]. In response, local private and community-based initiatives led to the establishment of small and medium-scale oil processing units, which now play an increasing role in cottonseed oil production [7].

According to FAOSTAT, Mali remains a major consumer of cottonseed oil, with an average consumption of about 2.56 kg per capita per year [3]. However, cotton cultivation relies heavily on pesticides, especially organophosphates, carbamates, and pyrethroids [8]. The presence of pesticide residues in cottonseed oil therefore raises concerns regarding food safety. Studies conducted in other cotton-producing countries, including Burkina Faso, Benin, Nigeria, and India, have reported pesticide residue levels in edible oils, sometimes exceeding international maximum residue limits [9–11]. However, few recent studies have addressed this issue in Mali, particularly in semi-industrial and artisanal oil production units.

Therefore, it is essential to evaluate the contamination levels of cottonseed oils produced locally in Mali in order to protect consumer health and support national food safety monitoring. The present work contributes to this effort and is part of laboratory analyses conducted on cottonseed oil samples submitted to the Environmental Toxicology and Quality Control Laboratory (ETQCL) in Central Veterinary Laboratory (CVL).

## 2. Materials and Methods

### 2.1. Research Design

This pilot study consisted of analyzing cotton oil samples

submitted for analysis to the Environmental Toxicology and Quality Control Laboratory (ETQCL) during November and December 2024. The objective was to identify and quantify pesticide residues in order to support marketing authorization applications submitted by oil mills and production units. The research followed a laboratory analytical protocol using gas chromatography equipped with a microelectron capture detector (GC- $\mu$ ECD), a standard method for determining organochlorine, organophosphorus, and pyrethroid pesticide residues in accordance with international guidelines [12, 13]. A list of nine organochlorine pesticides to be screened for in cottonseed oils was targeted (Table 1) and selected because of their potential for persistence despite their prohibition.

**Table 1.** List of Pesticides to Look for.

| Pesticide    | Group          |
|--------------|----------------|
| Aldrin       | Organochlorine |
| Dieldrin     | Organochlorine |
| Endosulfan A | Organochlorine |
| Endosulfan B | Organochlorine |
| Endrin       | Organochlorine |
| Heptachlor   | Organochlorine |
| Lindane      | Organochlorine |
| op'DDT       | Organochlorine |
| pp'DDT       | Organochlorine |

#### 2.1.1. Inclusion and Exclusion Criteria

Cotton seed oil samples submitted to ETQCL for pesticide residue testing are included in the analysis. However, some samples are excluded from this process due to insufficient volume, damaged packaging, or incomplete sampling information. This rigorous sample selection process ensures the reliability of results and compliance with safety standards.

#### 2.1.2. Sampling and Sample Sources

The samples for this pilot study were obtained through additional submissions made simultaneously with the original samples. These samples were provided directly by oil mills and cottonseed oil processing units, or through consulting firms selected to assist them in the marketing authorization process.

**Table 2.** Distribution of Samples Submitter.

| Submitting Type  | Number of Samples | Percentage (%) |
|------------------|-------------------|----------------|
| Consulting Firms | 20                | 74.07          |

| Submitting Type      | Number of Samples | Percentage (%) |
|----------------------|-------------------|----------------|
| Oil Processing Units | 07                | 25.93          |
| Total                | 27                | 100            |

## 2.2. Analytical Method

The determination of pesticide residues in cottonseed oil samples was performed using gas chromatography (GC- $\mu$ ECD), following four main steps in accordance with ISO guidance [14]: extraction, concentration, purification, instrumental analysis, and quantification. This rigorous method ensures the accuracy and reliability of the results obtained.

### 2.2.1. Liquid Liquid Extraction

2 g of oil sample were weighed, to which 20 g of anhydrous sodium sulfate were added, then mixed thoroughly to remove moisture. Once this step was complete, the mixture was transferred to a separating funnel. Next, sequential extraction was performed using 80 ml, then 150 ml of dichloromethane, while stirring to ensure proper extraction of the organic compounds. After this extraction, the organic phase was recovered and concentrated to approximately 2 ml using a rotary evaporator. Finally, this concentrate was transferred to a glass tube, rinsed with hexane, and the final volume was adjusted to 10 ml to obtain a sample ready for concentration and analysis.

### 2.2.2. Concentration of Extracts

The extract was concentrated using a rotary evaporator to reduce solvent volume and obtain a detectable analyte concentration.

### 2.2.3. Purification of Extracts

To prepare the extracts for gas chromatography (GC) analysis, 1 mL of sulfuric acid was added to break down the lipid content in the sample. This step is essential to remove lipids that could interfere with the analysis results. Once this degradation was complete, the mixture was concentrated to approximately 0.5 mL to obtain a more concentrated solution suitable for analysis. Finally, these purified extracts were transferred to 1.5 mL vials, ready for GC analysis. The intended impact of this step is to help ensure that samples are

properly prepared and optimized to obtain reliable results.

### 2.2.4. Instrumental Analysis

The Agilent 7890 gas chromatograph (GC) equipped with an electron capture detector (ECD) was used for instrumental analysis of the sample extracts. The required temperature and pressure conditions used are shown in the following table.

**Table 3.** Gas Chromatographic Conditions.

| Parameter                | Condition                                                                                     |
|--------------------------|-----------------------------------------------------------------------------------------------|
| Oven Initial Temperature | 70 °C                                                                                         |
| Temperature Program      | 70°C → 150 °C (25 °C/min), then to 200 °C (5 °C/min), then to 260 °C (10 °C/min, 15 min hold) |
| Carrier Gas              | Nitrogen, 1.6 mL/min                                                                          |
| Makeup Gas               | Nitrogen, 60 mL/min                                                                           |
| Injector Temperature     | 280 °C, splitless mode                                                                        |
| Detector Temperature     | 300 °C                                                                                        |

A mixed standard pesticide solution consisting of the nine organochlorine pesticides with a certified purity to be tested for and listed in Table 1 was prepared in hexane. A five-point calibration curve was generated at concentrations of 0.0125, 0.025, 0.05, 0.10, and 0.125  $\mu$ g/mL. Pesticides were identified by comparing the retention time of chromatographic peaks in samples to those of standard solutions injected under identical conditions. Quantification was based on peak area response relative to the calibration curve.

## 3. Results

Among the 27 cottonseed oil samples analyzed, 17 samples were free of the targeted pesticide residues, while 10 samples were contaminated with organochlorine pesticide residues. The concentrations of detected residues are summarized in Table 4. The frequency of pesticides detected, expressed as the number of times detected, is summarized in Figure 1.

**Table 4.** Contaminated Cottonseed Oil Samples.

| code | Pesticides Residues (mg/L) |          |              |              |        |           |        |        |
|------|----------------------------|----------|--------------|--------------|--------|-----------|--------|--------|
|      | Aldrin                     | Dieldrin | Endosulfan A | Endosulfan B | Endrin | Heptchlor | pp'DDT | Lindan |
| Ec1  | 0,082                      | ND       | ND           | ND           | ND     | ND        | ND     | ND     |
| Ec2  | 0,081                      | ND       | ND           | ND           | ND     | ND        | ND     | ND     |
| Ec3  | 0,107                      | ND       | ND           | ND           | ND     | ND        | ND     | ND     |

| code | Pesticides Residues (mg/L) |          |              |              |        |           |        |        |
|------|----------------------------|----------|--------------|--------------|--------|-----------|--------|--------|
|      | Aldrin                     | Dieldrin | Endosulfan A | Endosulfan B | Endrin | Heptchlor | pp'DDT | Lindan |
| Ec4  | 0,097                      | 0,097    | ND           | 0,187        | 0,085  | 0,110     | 0,244  | ND     |
| Ec5  | 0,097                      | 0,100    | ND           | 0,181        | 0,085  | 0,111     | 0,501  | 0,09   |
| Ec6  | 0,099                      | 0,095    | ND           | ND           | 0,086  | 0,116     | 0,221  | ND     |
| Ec7  | 0,098                      | ND       | ND           | ND           | 0,085  | 0,148     | 0,225  | ND     |
| Ec8  | 0,099                      | 0,096    | ND           | ND           | 0,084  | 0,154     | ND     | ND     |
| Ec9  | 0,094                      | 0,095    | 0,157        | ND           | 0,085  | 0,081     | ND     | ND     |
| Ec10 | 0,060                      | 0,046    | 0,097        | ND           | ND     | ND        | ND     | ND     |

The results of the analyses showed that around ten of the twenty-seven oil samples submitted were contaminated with pesticide residues. The analysis of 27 cottonseed oil samples revealed that 62.95% of the samples were free from the targeted organochlorine pesticides, while 37.05% were contaminated. Lindane was detected in a single sample at a concentration of 0.090 mg/L. The insecticides Endosulfan A and B were identified in two samples each, with concentrations ranging from 0.097 to 0.157 mg/L for Endosulfan A and from 0.181 to 0.187 mg/L for Endosulfan B. DDT was found in four samples, with concentrations ranging from 0.221 to 0.501 mg/L. In addition, Endrin and Heptachlor were detected together in the same samples, with Endrin concentrations ranging from 0.084 to 0.086 mg/L and Heptachlor concentrations ranging from 0.081 to 0.154 mg/L. Dieldrin was observed in six samples, with concentrations ranging from 0.046 to 0.100 mg/L. Finally, Aldrin was present in all ten contaminated samples, with concentrations ranging from 0.060 to 0.107 mg/L.

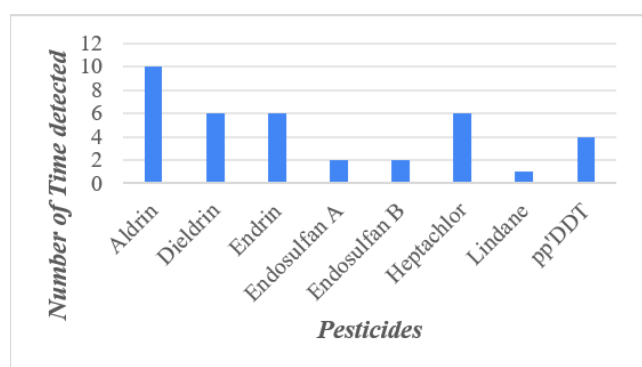


Figure 1. Frequency of pesticides detected.

## 4. Discussion

From the perspective of contamination prevalence, the fact that more than one-third of the samples (ten out of twenty-seven) were contaminated with pesticide residues is alarming. This indicates a significant level of pollution that could have

implications for the quality of locally produced cottonseed oil and, consequently, for consumer health.

This pilot study focused solely on certain organochlorine pesticides, whereas in Mali cotton production involves the use of three main pesticide classes: organochlorines, organophosphates, and carbamates [15]. Consequently, the designation of “non-contaminated” samples should be interpreted with caution, as residues of carbamates could still be present, consistent with previous observations in Mali and other West African countries [16]. The time elapsed between pesticide application and oil processing likely contributed to the absence of detectable organophosphates in the analyzed samples. The time elapsed between pesticide application and oil processing likely contributed to the absence of detectable organophosphates in the analyzed samples.

Among the nine organochlorine pesticides targeted, op' DDT was not detected in any sample, whereas the remaining eight were present at varying concentrations. pp' DDT exhibited the highest residue levels, reaching 0.501 mg/L, while dieldrin showed the lowest concentration at 0.046 mg/L. These findings are in line with reports from West Africa, where persistent organochlorines such as DDT and endosulfan remain detectable in cotton-derived products due to historical use and environmental persistence [17, 18].

The detection of multiple organochlorine residues in the same samples also reflects the widespread and cumulative use of these chemicals over time. Importantly, several detected pesticide levels exceeded the maximum residue limits (MRLs) for edible oils, indicating potential risks to human health. Similar exceedances have been reported in neighboring countries, such as Burkina Faso and Benin, where organochlorine residues in locally produced oils frequently surpass regulatory thresholds [19, 20]. These results highlight the urgent need for stricter regulatory monitoring, the inclusion of all major pesticide classes including carbamates and organophosphates and public awareness programs to ensure the safety of cottonseed oil intended for consumption.

Overall, while the majority of samples appeared free from organochlorine residues, the presence of high levels in a substantial fraction underscores the persistence of these chemicals

and the necessity for comprehensive pesticide residue monitoring in Mali and other cotton-producing regions.

## 5. Conclusions

This pilot study provided baseline data on organochlorine pesticide contamination in cottonseed oils produced in Mali. Among the ten targeted compounds, eight were detected, with some exceeding the maximum residue limits, particularly DDT. Such contamination poses potential health risks, emphasizing the importance of monitoring even at low residue levels. These findings raise critical questions about food security and public health in Mali. Proactive action is needed to address this worrying situation. To ensure consumer safety, it is essential to implement measures that reduce the use of persistent pesticides in cotton cultivation, adopt good agricultural and processing practices, and establish routine residue monitoring programs. Promoting awareness among farmers and processors and exploring safer alternatives will further help minimize pesticide transfer to edible oils.

## Abbreviations

|         |                                                               |
|---------|---------------------------------------------------------------|
| AOAC    | Association of Official Analytical Chemists                   |
| CMDT    | Malian Textile Development Company                            |
| CVL     | Central Veterinary Laboratory                                 |
| DDT     | Dichloro-Diphenyl-Trichloroethane                             |
| ECD     | Electron Capture Detector                                     |
| ETQCL   | Environmental Toxicology Quality Control Laboratory           |
| FAOSTAT | FAO's Global Statistical Database                             |
| GC      | Gas Chromatograph                                             |
| GDP     | Gross Domestic Product                                        |
| MRLs    | Maximum Residue Limits                                        |
| ND      | Not Detected                                                  |
| HUICOMA | Mali Cotton Oil Mill                                          |
| INSTAT  | Mali National Institute of Statistics                         |
| ISO     | International Standardization Organisation                    |
| USTT-B  | University of Sciences, Techniques and Technologies of Bamako |

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## Data Availability Statement

The data is available from the corresponding author upon reasonable request.

## Conflicts of Interest

The authors declare no conflicts of interest.

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