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A Qualitative Study of TLC for Detection of Pesticides and Poisons in Food Products

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ABSTRACT:

In agriculture, pesticides are usually meant for protecting crops from pests and infections; however, their interaction with different toxic substances during forensic assignments has not been explored that much. The present work aims at throwing light on the chemical interference between selected pesticides (Dimethoate, Chlorpyrifos, Imidacloprid) and other toxic substances (Zinc Phosphide) present in food products. The samples studied are Fruits (Sapodilla, Indian Gooseberry, Pomegranate, Banana, Lemon) and Vegetables (Carrot, Onion, Green chili, Spinach, Tomato). The objective of the study is to find out the transformation or degradation of pesticide residues and thereby affect Rf value by these interactions. The Thin layer chromatography (TLC) analysis will be carried out using silica gel plates with optimized solvent systems (Acetone, Hexane, Ethyl Acetate, Methanol). The visualizations following separation will be done under UV lights. Changes in Retention Factors (Rf) values and the behavior of compounds are to be analyzed so as to determine chemical interactions. The initial results are likely to show a significant shift in the Rf values due to open hydrolysis, complex formation, or oxidation of pesticide molecules in the present conditions of toxins. The outcome obtained will be extensive knowledge pertaining to forensic toxicology, food safety monitoring, and analytical chemistry, by providing an affordable and efficacious technique that can ascertain pesticide-poison interactions within real-life scenarios and to determine whether pesticide-poison interactions lead to new detectable compounds.

Keywords: Poisons, Pesticides, TLC, Rf values, Food components, Chemical interactions.

INTRODUCTION:

TLC detection was evaluated for the applicability in a cost-effective means of screening pesticides and poisons through the performance of certain experiments. Here, a fast, sensitive application of thin-layer chromatography for detecting and measuring residues of different pesticides and poisons was prepared [3]. Regard the identification of toxic residues like pesticides and poisons in food, such an act entails an essence to public healthiness and safety [20]. Thin-layer chromatography (TLC) provides a cost-effective and simple way to analyze the entry of pesticides and poisons into complex matrices of food. Validation experiments were conducted on standard toxic compounds spiked into some relevant food samples. Optimized TLC method provided enhanced limits of detection, reduction in matrix effects, and the simultaneous separation of multiple analytes [23].

Pesticides are extensively used in agriculture, but their ability to interact with any other toxic agents like Imidacloprid, Chlorpyrifos, Dimethoate, Zinc phosphide remains unexplored in forensic toxicology. Such interactional studies can potentially lead to the formation of decomposed or toxic derivatives and certain alterations in the Rf values in TLC.

The study aims to highlight such interactions and their influence on pesticide residues in both food and forensic samples [25].

Poison is any substance (solid, liquid, or gas) that, when introduced into the body, can deteriorate health or cause death. The difference between medicine and poison lies in dosage and intent—medicine in excess can be toxic, while a poison in small amounts may have medicinal effects. Most poisoning cases occur at home, involving household products like cleaning agents, fuels, medicines, and cosmetics. Some animals produce venom, while others carry harmful bacteria. Additionally, certain household plants can be toxic to humans and animals [17].

Any substance or mixture of substances intended for preventing, destroying or controlling any pest, including vectors of human or animal disease, unwanted species of plants or animals, causing harm during or otherwise interfering with the production, processing, storage, transport, or marketing of food, agricultural commodities, wood and wood products or animal feedstuffs, or substances that may be administered to animals for the control of insects, arachnids, or other pests in or on their bodies. Thin Layer Chromatography (TLC) is an effective analytical technique for separating and identifying components in pesticide formulations. The separation efficiency in TLC depends on various factors, including the chemical nature of the analytes and the choice of stationary and mobile phases [18].

Chlorpyrifos is Used to control a variety of pests in agriculture (e.g., termites, mosquitoes, and crop-damaging insects). Work by inhibiting acetylcholinesterase, leading to nervous system failure in insects. Highly toxic if ingested, inhaled, or absorbed through the skin. Symptom of poisoning: Nausea, dizziness, respiratory distress, convulsions, and even death [8].

Imidacloprid is Used against sucking pests (aphids, whiteflies, termites) in crops and gardens. Systemic insecticide absorbed by plants and transferred to pests through ingestion. Moderately toxic to humans but highly toxic to bees and aquatic organisms. Symptoms of poisoning: Dizziness, respiratory distress, nausea, muscle weakness, and severe cases can lead to coma [14].

Dimethoate is used to Broad-spectrum systemic and contact insecticide used in cotton, wheat, vegetables, tea, and fruit crops. Effective against aphids, thrips, Jassi's, whiteflies, and mites. Binds to and inhibits acetylcholinesterase (AChE), preventing the breakdown of acetylcholine. Leads to continuous nerve signal transmission, causing paralysis and death in insects. It was highly toxic (WHO Class II). Symptoms of poisoning include nausea, vomiting, muscle weakness, convulsions, blurred vision, respiratory distress, and even coma in severe cases [9].

Zinc Phosphide is used to kill rats, mice, and other rodents in agriculture, warehouses, and urban areas. Upon ingestion, reacts with stomach acid (HCl) to release phosphine gas (PH_3). Phosphine gas disrupts mitochondrial function, leading to respiratory failure and organ damage. Causes nausea, vomiting (black or bloody), breathlessness, convulsions, cardiovascular collapse, coma, and death. Delayed toxicity: Death usually occurs within 24-48 hours due to multiple organ failure [16].

Objective of this study is to detect and identify specific poison or harmful chemicals present in food sample and to analyze the changes in TLC separation patterns (Rf values) of Dimethoate, Imidacloprid, Chlorpyrifos upon interaction with poisons (Zinc phosphide). To determine whether pesticide-poison interactions lead to new detectable compounds.

MATERIALS AND METHOD:

Samples and Materials:

Samples are Food samples like Fruits (Sapodilla, Indian Gooseberry, Pomegranate, Banana, Lemon) and Vegetables (Carrot, Onion, Green chili, Spinach, Tomato). Selected Pesticides are Chlorpyrifos, Imidacloprid, Dimethoate and Selected Poison is Rat poison (Zinc phosphide).

The study was conducted using essential laboratory apparatus and safety measures to ensure accurate analysis and safe handling of chemicals. A Thin Layer Chromatography (TLC) chamber and silica gel TLC plates (GF254) were used for separation. Samples were prepared using a mortar and pestle, stored in labeled containers, and covered with aluminum foil to prevent contamination. Micro capillary tubes, 10 ml beakers, droppers, pencils, and measurement scales were used for precise sample application and measurement. Developed plates were handled with forceps and observed under a UV chamber for detection. Solvents such as ethyl acetate, hexane, methanol, and acetone were utilized to prepare the mobile phase and extract pesticide residues.

Throughout the experiment, safety equipment including lab coats, gloves, safety goggles, and face masks were worn to protect against chemical exposure and maintain laboratory safety standards.

METHODOLOGY:

Sample Preparation:

The Fruits and Vegetables are collected in local Provision store and Fruits shop and they are washed and cleaned before start the extraction procedure. 5g of samples taken and they are chopped well. Then the samples are soak in the pesticides (2ml) and the poison (2g) in a beaker. Then it was leave in one hour at room temperature. (fig1)

Fig: 1 Sample preparation

Sample Extraction:

The samples are crushed in Mortar and Pestle and add 2ml of Acetone into a paste consistency. Then it was stored in a container at room temperature (fig 2).

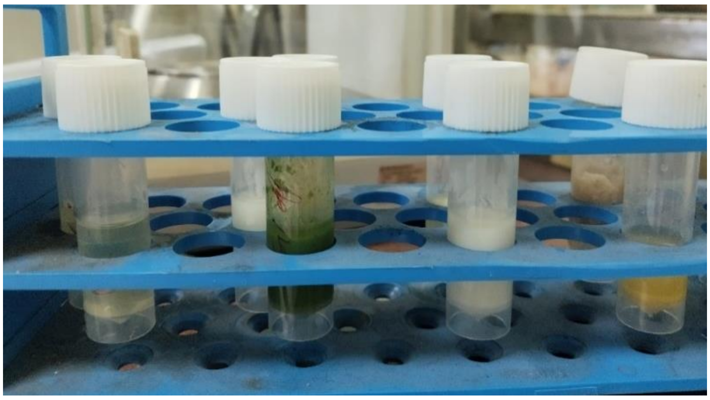

Fig: 2 Samples are stored in a Containers

TLC Preparation:

Solvent preparation:

The Mobile Phase is the component that helps the analyte to interact with the stationary phase. For this research, a solvent system composed of Ethyl acetate, Hexane, Methanol in a ratio of 7:3:1 was used due to its efficiency in separating and identifying the compounds present in the Pesticides and Poison.

Procedure:

Place the solvent system (i.e., ethyl acetate: hexane: ethanol) in the proportion of 7:3:1 in the TLC chamber. Prepared food samples extractions are spotted on the pre-coated silica gel TLC plate about 2 cm above the bottom of the plate with the help of capillary tubes.

Food samples extractions are spotted to TLC plate, sized (5 × 10 cm). The plate was placed in the developing chamber that contained the solvent system (mobile phase). It took 30-45 minutes for the solvent system to reach the top of the plate. Once the solvent traveled within a top of the plate or 3/4 in the TLC plate, the plate was removed from the chamber. The solvent front and the chromatographic spots were observed under UV Chamber and marked using a pencil and recorded as a photograph under UV light. Finally, the qualitative analysis of thin-layer chromatography was measured by calculating the respective retention factor of each spot obtained in the plate (fig 4a, fig4b).

Fig: 3 TLC Chamber and Silica gel Plate

RESULIT AND DISCUSSIONS;

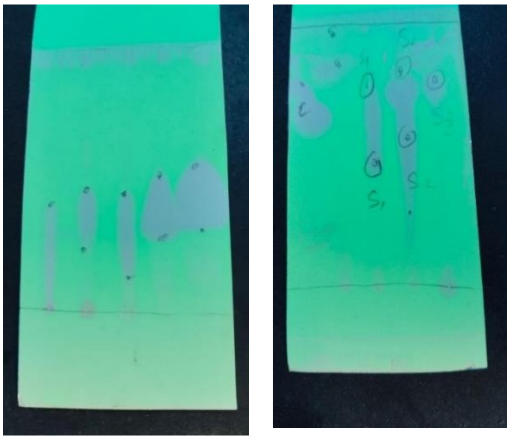

Fig: 4(a) Visualizing under TLC plate in UV Chamber

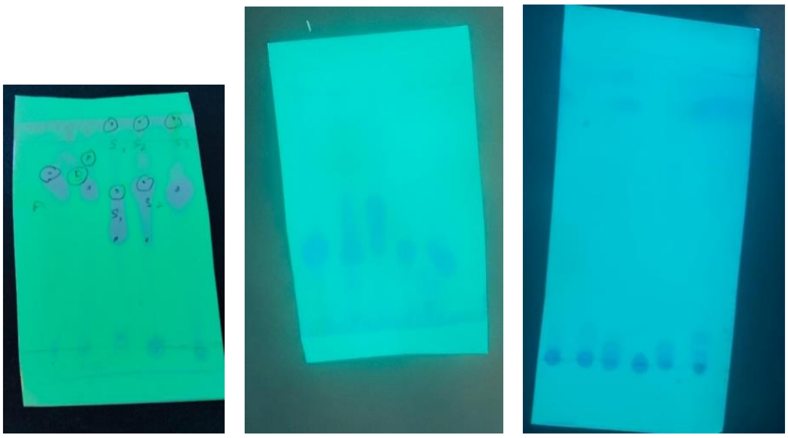

Fig: 4 (b) Visualizing under TLC plate in UV Chamber

Table: 1 Standard values of the Pesticides and Poison [22,24]

S. No	Name	Sample Name	Standard value	Experimental Value
1	Chlorpyrifos	C1	0.52 – 0.58	0.60
2	Imidacloprid	C2	0.35 – 0.42	0.45
3	Dimethoate	C3	0.45 – 0.50	0.52
4	Zinc Phosphide	C4	0.10 – 0.15	0.10

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Table: 2 Rf Value Calculations from Samples Analysis

S. No	Name	Sample Name	Imidacloprid (C2)	Dimethoate (C3)	Poison mixture (C1+C4)	Double pesticide Mixture Value (C2+C3)
1	Sapodilla	S1	0.40	0.46	0.63	0.68
2	Indian Gooseberry	S2	0.33	0.40	0.67	0.77
3	Pomegranate	S3	0.42	0.48	0.63	-
4	Banana	S4	0.38	0.49	0.65	0.65
5	Lemon	S5	0.30	0.40	0.65	0.65
6	Carrot	S6	0.39	0.53	0.66	0.66
7	Onion	S7	0.35	0.51	0.65	0.65
8	Green chilly	S8	0.33	0.43	0.60	0.47
9	Spinach	S9	0.36	0.48	0.52	0.52
10	Tomato	S10	0.40	0.50	0.66	0.56

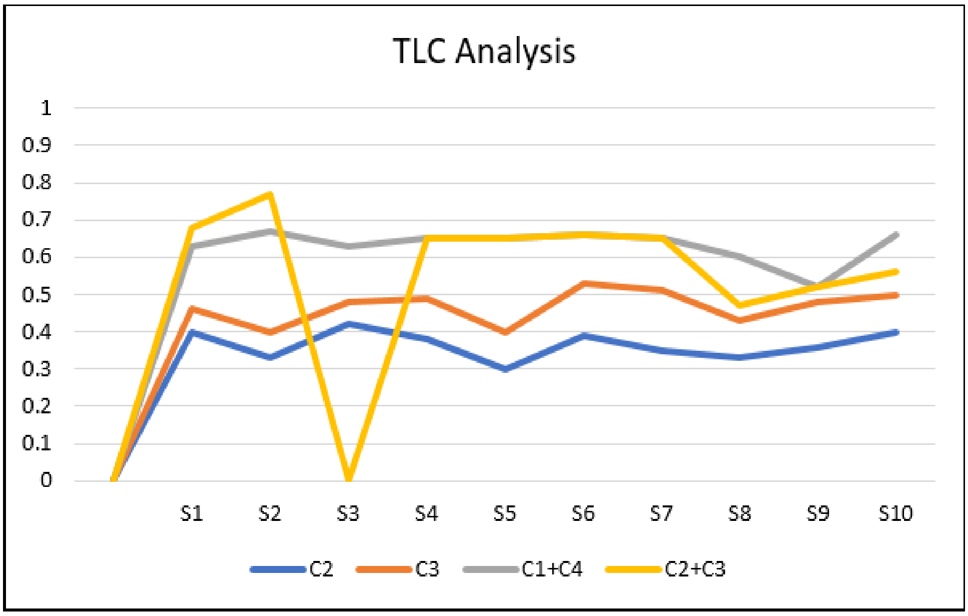

Fig: 5 Graphical representations of Rf Value Calculations from Samples Analysis

Food components interactions and Major components:

Table :3 Major Compound of Sample and Interactions [12,13]

Name	Sample Name	Major components of food samples	Interactions of food components with Pesticides	Interactions of food components with Poison
Sapodilla	S1	Sugars, Tannins, Polyphenols	Tannins may bind to pesticides, altering solubility and mobility. Sugars might promote slight hydrolysis.	Polyphenols may react with zinc phosphide, potentially affecting detection
Indian Gooseberry	S2	Vitamin C, Polyphenols	High acidity may cause partial degradation of dimethoate, reducing recovery efficiency.	Acidic nature may accelerate zinc phosphide breakdown, forming phosphine gas.
Pomegranate	S3	Anthocyanins, Tannins	Anthocyanins may interact with imidacloprid, leading to pigment-pesticide complex formation.	Acidic pH may trigger hydrolysis of zinc phosphide, altering its retention.
Banana	S4	Starch, Flavonoids	Starch may absorb pesticides, leading to reduced separation efficiency in TLC.	Zinc phosphide may show delayed breakdown due to lower acidity.

Lemon	S5	Citric Acid, Flavonoids	Citric acid may accelerate hydrolysis of dimethoate. Essential oils could modify solubility in the mobile phase.	Strongly acidic nature enhances decomposition of zinc phosphide into toxic phosphine gas.
Carrot	S6	Carotenoids, Sugars	Carotenoids may create pesticide-lipid interactions, affecting mobility. Sugars may cause mild binding.	Zinc phosphide interaction is minimal, but fiber content may retain traces.
Onion	S7	Quercetin, Sulfur Compounds	Sulfur compounds may chemically alter pesticide structures, causing degradation	Sulfur interactions might modify zinc phosphide breakdown, leading to unpredictable behavior.
Green chilly	S8	Capsaicinoids, Flavonoids	Capsaicinoids may bind to pesticides, affecting their polarity and TLC migration.	Minimal direct interaction, but capsaicinoids may affect extraction efficiency.
Spinach	S9	Chlorophyll, Flavonoids	Chlorophyll may form complexes with imidacloprid, modifying Rf values. Oxalates may react with dimethoate, reducing its mobility.	Zinc phosphide interactions could be limited, but oxalates may cause precipitation effects.
Tomato	S10	Lycopene, Carotenoids	Organic acids may accelerate dimethoate hydrolysis, reducing detectability. Lycopene may interfere with pesticide binding.	Acidic pH facilitates zinc phosphide breakdown, forming phosphine gas.

DISCUSSIONS:

The study identified several types of interactions between food components and pesticides that significantly influenced TLC analysis. Acidic foods like lemon, tomato, Indian Gooseberry, and pomegranate promoted hydrolysis of dimethoate and imidacloprid, forming more polar degradation products such as O, O-dimethyl thiophosphate, formaldehyde, and 6-chloronicotinic acid, which led to decreased Rf values. Imidacloprid also formed complexes with polyphenols and flavonoids found in spinach, onion, and green chili, resulting in new TLC spots or reduced intensity of the original pesticide signal. Dimethoate underwent sulfur-induced degradation when exposed to sulfur compounds in onion, producing O, O-dimethyl phosphorothioate and causing poor TLC separation [17]. Zinc phosphide reacted with citric acid in acidic foods like lemon and pomegranate, releasing toxic phosphine gas, which was not detectable via TLC but posed serious health risks. Oxidation reactions between dimethoate and carotenoids in carrot and tomato slightly increased Rf values due to reduced polarity. Additionally, imidacloprid formed binding complexes with starch in banana and sapodilla, and with chlorophyll in spinach, both of which hindered extraction efficiency and reduced TLC detectability. These interactions highlight the complex chemical behavior of pesticides in food matrices and their impact on TLC analysis [17].

CONCLUSION:

The research underlines the mode of action by which food naturally occurring compounds, e.g., organic acids, flavonoids, sugars, and sulfur-containing compounds, influence pesticides like Imidacloprid, Dimethoate, and Zinc Phosphide, altering their Rf values upon Thin Layer Chromatography (TLC) analysis. These involve competing adsorption, chemical breakdown, acid- or enzyme-induced conversion, and co-elution for the case when there are various pesticides present together. Acidic foods (such as lemon and pomegranate) favor degradation, whereas sugary or starchy foods hinder pesticide mobility [2]. Polyphenol- and sulfur-containing foods may bind to or chemically modify pesticides, leading to erratic retention behavior and making detection difficult.

Because of these matrix effects, pesticides frequently do not retain their characteristic Rf values in actual food samples. This illustrates TLC's value as a screening tool, but also its shortcoming in complex matrices. The research suggests sophisticated methods such as HPLC or GC-MS for precise pesticide identification and quantification in forensic and food safety laboratories. It also tested repeatability and Minimum Detectable Quantities (MDQ) for different groups of pesticides, affirming that food constituents greatly affect TLC outcomes and need to be taken into account during analysis [21].

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Conflict of Interest Statement:
The authors have no conflicts of interest to declare.