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Original Research Article

Analysis and detection of metals in synthetic food colors of sweets by TLC, FTIR and AAS

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ABSTRACT

Aim: To determine the presence of heavy metals (Pb,Cd,Co and Ni) in food colors using qualitative and quantitative methods.
Objective: Qualitatively asses food color sample purity through TLC. Quantitatively analyze heavy metal content by AAS and identify functional groups by FTIR.
Background: Food adulteration is a global public health concern, with synthetic food colors often used, including non permitted variants. Study aimed to address this issue by analyzing 15 samples from local markets for heavy metal presence in food colors.
Material and Methods: 15 samples collected from local markets including sweets, sweet drops, juices and gelatin, standard food color samples. Chemicals used Butanol,
GAA, kbr,D/W. TLC chamber, FTIR, AAS equipment were used along with standards of Pb, Cd,Co and Ni.
Results: TLC analysis revealed synthetic food colors in most samples FTIR identified functional groups in Samples and AAS detected metal presence in 2 out of 15 samples.
Conclusion: In summary, the study revealed that many local market food color samples contain synthetic colors and traces of harmful heavy metals like cadmium and Cobalt.

1. Introduction

Food color or food dyes constitute a crucial group of additives, encompassing pure substances derived either naturally from raw materials or synthetically produced. Synthetic dyes exhibit greater stability in food products compared to natural colorants.[1–3] Pleasant colors in food and beverages captivate young children's attention. Many unpackaged edibles, including snacks, bakery items, candies, and beverages, contain banned colors and toxic chemicals.[2] Growing evidence indicates potential adverse effects stemming from dye overuse[3–5] Applying natural food colors demands prudent and informed usage, necessitating precise characterization of color substances. Scientific communities continually update color-related information based on research outcomes, almost on an annual basis[4–7] FDA regulations mandate certification for synthetic food colors, whereas natural food colors are exempt[8,9] Per the Food Adulteration Act (1954), permitted food colors encompass three red shades (Carmoisine, Ponceau 4R, Erythrosine), two yellow shades (Sunset yellow FCF, Tartrazine), two blue shades (Brilliant blue FCF, Indigo Carmine), and one green shade (Fast green FCF). However, adulterants such as Metanil yellow, Rhodamine B (RB), Orange G, and Sudan dyes are illicitly introduced into food[10] The identification and quantification of color additives in food utilize Thin layer chromatography, Fourier transform infrared spectroscopy, and Atomic absorption spectroscopy.[11,12]

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2. Materials and Methods

1. Samples Collected: A total of 15 samples were obtained, comprising 3 samples of small sweet drops (boondi), 3 sweet samples, 5 juice samples, and 4 gelatin (jelly) samples, all procured from the local market, alongside food color standards.
2. Chemicals and Reagents: The following chemicals and reagents from Merck, Qualikems, AR/ACS, NA 65% were employed: TLC plate, butanol, glacial acetic acid, nitric acid, KBr, and triple distilled water.
3. Equipment Used: The equipment utilized included a TLC chamber, Fourier transform infrared spectroscopy, and atomic absorption spectroscopy.
4. Standard Preparation: A standard solution of 1 ppm was prepared using AAS standard solution with a concentration of 1000 μg/ml for each metal (lead, cobalt, cadmium, and nickel).
5. TLC: Thin layer chromatography is a method used to determine the number components in a mixture. Verify the identity the purity of a compound. In TLC the stationary phase is a thin adsorbent material layer coated with silica gel the sample is spotted on the one end of the TLC plate and placed vertically into a closed chamber with an organic solvent (mobile phase).

Fourier transform infrared spectroscopy(Figure 1) is an analytical technique use for the judgement of Product authenticity and quality and helps to identify the content of sugars, pigment, organic acid, volatile, phenolic compounds and functional groups present in sample.[13]

Fig. 1: Fourier transform infrared spectroscopy perkin FTIR system spectrum BX

Atomic Absorption Spectroscopy (Figure 2) Is a technique for determining the concentration of a particular metal element in a sample,[14] Elemental analysis is important for productlabelling and quality control[14,15] Use for the detection of elements such as mercury, Lead, Cadmium, Cobalt etc.

2.1. Procedure

TLC:- A qualitative analytical method, was applied to synthetic food colors in the 15 samples, encompassing 3 types of small sweet drop samples (boondi), 3 types of sweet samples (mithai), 4 types of gelatin samples (jellies), and 5 types of juice samples. Each 5 g of sample was dissolved in 5 ml of glacial acetic acid. The mixture, along with standard food colors, was applied to a pre-coated TLC plate and separated using a mobile phase (Butanol 2.5 ml, Glacial acetic acid 1.5 ml, Water 1.5 ml) [2.5:1.5:1.5]. Visualization of spots was performed under an iodine chamber.

Fig. 2: Atomic Absorption Spectrophotometer Electronics of India Ltd Element AS AAS 4141

FTIR:- For Fourier transform infrared spectroscopy analysis, 5 g of each sample was placed in a petri dish and subjected to drying in a hot air oven at 100°C for 3-4 days, with intermittent intervals to eliminate moisture content. The dried samples were then mixed with a small amount of KBr to create pellets suitable for analysis.

AAS:

1. Acid digestion method- All the 15 samples along with food color standards weigh at 5 gms/ml to dissolve in 10 ml nitric acid left for 2 days. After two days samples were boiled on heating instrument for completely dissolved. In this method some samples like jelly, Boondi and mango juice were not dissolved so they were proceed by another method i.e.
2. Dry Ashing method – in this method samples were kept in muffle furnace at 700°C for 3 hours. After that the Ash of each sample dissolved in 5 ml nitric acid left it for 1 day and after that filter it with watmann filter paper to remove the particles. Make up the obtained extract with triple distilled water in 50 ml of volumetric flask

3. Results and Discussions

Table 1: Rf value of food color samples by using TLC

Sample type	Mean	Median	Mode	Range
Sweets(n=3)	0.58	0.44	0’44	0.93-0.21
Small sweet drops (n=3)	0.556	0.44	0.34	0.82-0.34
Juices(n=5)	0.62	0.615	NIL	0.41-0.86
Gelatin(n=4)	0.56	0.53	NIL	0.41-0.71

Table 1 shows the mean, median, mode and rangefor all the 15 samples as this is the combine result of all the 15 samples.

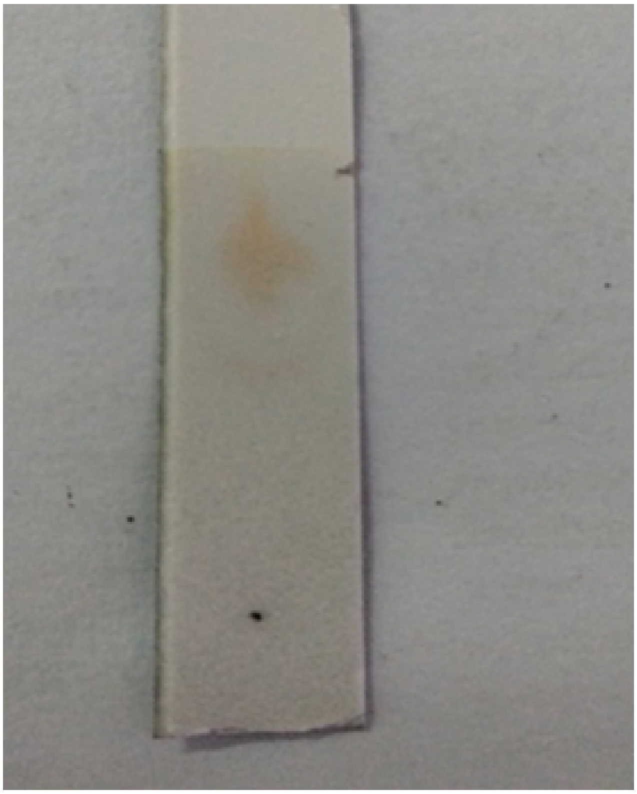

Fig. 3: TLC image

Table 2: Functional group peaks obtained using FTIR

Functional groups	Sweets (n=3)	Sweet drops (n=3)	Juices (n=5)	Gelatin (n=4)
Aldehyde	2	3	2	nil
Carboxylic Acid	3	2	5	4
Alkene	2	2	Nil	4
Carbonyl compound	2	1	1	1
Amino group	2	3	3	4
Alkane	1	2	5	4

Table 2 Shows the statistical data for all the 15 samples in which the number shows that which type of functional group is present in (n) number of sample.

Table 3 of Atomic absorption spectroscopy shows statistical result of all the 15 samples in which metal concentration of each sample can be calculated.

This study focused on detecting the metals in food colors, for which food beverages were chosen as samples. A total of 15 samples were taken, while 5 food colors

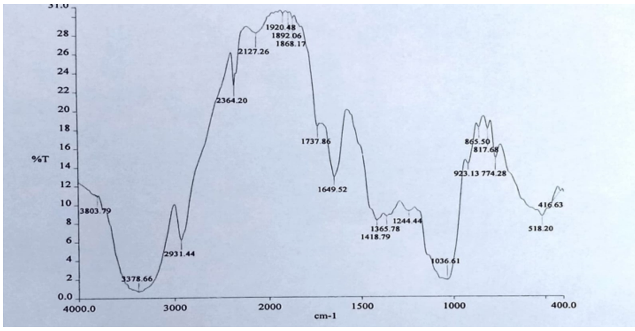

Fig. 4: FTIR spectra of statistical data shows peak ranges of FTIR

Table 3: Amount of metal obtained in each sample by using AAS

Sample Type	Lead concentration	Cadmium concentration	Cobalt concentration	Nickel concentration
Sweets(n=3)	0.018	0.030	0.238	0.000
Smallsweet drops(n=3)	0.018	0.030	0.064	0.000
Juices(n=5)	0.018	0.030	0.111	0.025
Gelatin(n=4)	0.018	0.030	0.145	0.033

were chosen to be compared with the samples. Different extraction procedures were applied to extract the food color-containing metal from the samples in this study.In a previous study, Kourani et al. demonstrated that sweet samples were dissolved in acetic acid (jalebi and laddu)[16]. In this study, all samples were dissolved in glacial acetic acid for TLC, and for Atomic absorption spectroscopy, all samples were treated with nitric acid. Results showed in this study that synthetic food colors were found in most of the samples through the TLC method, such as carmoisine, orange G, brilliant blue, erythrosine, and sunset yellow, according to the calculation of their RF values.In the previous study, Kourani et al. reported that only one shop from local and branded vendors contained prohibited dye (Metanil Yellow), while the rest yielded a negative result[16]. In this study, TLC showed a positive result for mithai, jelly, juice, and boondi, and somehow FTIR shows adequacy, and in Atomic absorption spectroscopy, (cd) and (co) metals were detected in 2 samples.

4. Conclusion

The study was done to identify the presence or absence of metal in food colors. All 15 samples collected from local market included different colors of sweets(mithai), small sweet drops(boondi), juice and Gelatin(jellies) contained synthetic colors and metals like cadmium (cd) and cobalt (co) were found present after performing quantitative analysis. The study revealed that food colors contains metal which causes toxicity or poisoining in living organism if found present in excess quantities.

5. Source of Funding

None.

6. Conflict of Interest

None.

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