
ORIGINAL ARTICLE

**Detection and Quantification of Adulterants in Food items
Purchased from Local Stores in Kashmir Valley of Jammu and
Kashmir**

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ABSTRACT

The present study on "Detection and Quantification of Adulterants in Food items purchased from local stores in Kashmir Valley of Jammu and Kashmir" was conducted in the department of bio resources and department of food technology University of Kashmir from April 2022-July2022. The objective of the present work was to investigate the adulteration in food items. The items selected for the study were chosen which form the most common consumed and basic food products in households. These selected products included spices and honey. The tests were done for the locally available products and from the origin supplier. It was concluded that all of the basic food items were found to be adulterated with at least one adulterant. The adulteration as concluded from the study has been done preferably to gain the profit margins in the market. It is recommended that the Govt. should take precautionary steps and tight hold over the testing bodies to ensure an adulteration free product in market. This will be an aiding step for achieving "Suvast Bharat Mission" of Govt. of India.

Keywords: Food adulteration, Detection, Quantification, local stores, awareness, Kashmir.

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INTRODUCTION

Food is stuff of either plant or animal origin which in raw, processed or semi-processed form will be taken in to the body in order to support the different biochemical and physiological activities of our body. Most of the times, these foods are prone to food fraud and adulteration, and put health disorder to consumers [1]. The term food fraud encompasses the deliberate substitution, addition, tampering or misrepresentation of food, food ingredients or food packaging, or false or misleading statements made about a product for economic gain [2]. The specific type of fraud is the fraudulent, addition of fake substances or removal or replacement of genuine substances without the purchaser's knowledge for economic gain by the seller. Though traditionally, people cook and eat foods with health process and incidents at home, in modern times, change in life styles and raise in income leads more and more peoples to have ready to eat foods at restaurant on regular basis [3]. The food in numerous of these outlets may have been cooked with poor quality constituents while still attract and satisfy the palate rather than provide a wholesome nutritional meal [4] Nevertheless, normally most people do not know these foods are adulterated by 25 to 30 percent as implied by the above authors. This causes a major impairment for the health these consumers. Quality and safety of food including the factors affecting them have major rising areas within the food supply chain and attracted attentions of researchers, government and regulatory bodies [5]. Food adulteration is one among these different emerging areas of science. The word adulteration can be viewed in different ways, either mixing or substitution of inferior substances to the one which is superior to the adulterants or removal of some of valuable constituents from a given food products [6]. The term adulterated in legal term is used to

mean that a food product fails to meet federal or state standards. Adulteration can also be the addition of a non-food item to increase the quantity of the food item in raw or prepared form, preservation, purpose, and improvement of appearance which can be done either intentionally or unintentionally [7]. Also included are any poisonous or deleterious substances which may render the food injurious to health. Consumers are always either at least sufferers of being cheated or even victims of disease as a result of adulteration of food stuffs and death to the worst. Therefore, it is crucial for consumers to be aware of common food categories that are contaminated, common adulterants, and the health effects of such adulterants [8]. Adulterants are defined as chemicals that should not be present in a particular food or beverage but may be purposefully added to more expensive ingredients to raise their apparent quantities, lower their manufacturing costs, or for some other fraudulent or malevolent reason [9]. Because of inadequate or non-existent government oversight, the act of food adulteration was more prevalent in cultures from very early times. The act of adulteration can occasionally involve extremely harmful chemicals and toxins. The practice of putting toxic chemicals in food products not only has an effect on the health of customers but also ruins the nutritional value of food [10]. Other than the fact that there is less recorded literature and research in the area as a result of several challenges connected to awareness, resources, attention, and others, the issue of adulteration in our country is equally as serious as it is in other countries [11].

MATERIAL AND METHODS

The present investigation entitled "Detection and Quantification of Adulterants in Food items purchased from local stores in Kashmir Valley of Jammu and Kashmir" was carried out during April 2022-July 2022 in the Department of bio resources and department of food technology University of Kashmir. The details of material used; methods adopted followed during the course of this investigation are described below:

Experimental Material

The experimental material consisted of samples of Honey, Spices (Chilli and Turmeric).

Sample Collection

The materials were collected from local stores of Batamaloo, Hyderpora and Hazratbal area of District Srinagar.

Experimental details

Honey

The quantitative analysis of various honey samples (in total 3) two were purchased from the local market of Srinagar, J&K and one from the supplier in Kashmir region were tested for presence of sugars, HMF and moisture content. Spectrophotometer, refractometer was employed for testing. Apart from that other chemicals were analytical grade (purchased from MERK). Following procedure was used for determination of different mentioned constituents.

- Refractometry
- High Performance Liquid Chromatograph (HPLC).
- Spectrophotometric measurements.

Determination of Moisture Content

The detection of moisture content was done in accordance with the method presented by Association official Analytical Chemists. An Abbe honey refractometer test was conducted at 20 Degree Celsius. The prism of the refractometer was cleaned and dried before the testing. The honey sample was put on the prism and refractive index was estimated and compared to the values from Chattaway's data.

Determination of HMF

Quantitative analysis of HMF in honey was carried as per the method outlined by White, J. W. Carrez solution-I and Carrez Solution – II was prepared by dissolving 15g analytical grade potassium ferrocyanide and 30g zinc acetate in water and diluting to 100mL. Bisulfide solution (0.2%) was prepared by dissolving sodium bisulfide in water. Next, a sample of honey 3g was transferred to 50mL volumetric flask with total of 25mL water. 0.5mL of each. Carrrez I & II solutions were added and mixed and diluted to volume. The mixed solution was filtered through filter paper and first 10mL of filtered solution was rejected. 5mL of remaining filtered solution was transferred to two test tubes and 5mL of distilled water was added to one (referred to as sample) and 5mL of prepared bisulfide solution to other (reference). Finally, absorbance of sample against reference was evaluated at 284nm and 336nm in 1cm cell and using absorbance values HMF was calculated using formula

$$\text{HMF (mg/100g honey)} = (A_{284} - A_{336}) * 24.95 * 3$$

Determination of Fructose to Glucose ratio (F/G)

Quantification of fructose and glucose in honey was done as per the following method

The preparation of sugar standards was done by dissolving 25mL methanol and 40mL distilled water in 100mL calibrated flask. The standard amount as designated by Bogdanov, was added accordingly. For

fructose determination 2g analytical grade fructose was added for glucose determination 1.5 g analytical grade glucose was added. The solutions were filtered using filter paper and transferred to vials. Now, the samples to be tested were prepared by adding 4g honey sample to 40mL distilled water and adding 25mL methanol to it in a 100mL flask. The solution was mixed and filtered by filter paper and transferred to vials. The column temperature was set at 30 deg Celsius and 10uL of samples were used for testing. The reference samples were first tested for peak lengths and corresponding honey sample was tested for peak lengths. The percentage of fructose and glucose in the sample was evaluated as

$$W(\text{g}/100\text{g}) = \frac{A1 \cdot V1 \cdot m1 \cdot 100}{A2 \cdot V2 \cdot m0}$$

A1- peak height of sample solution
 A2- is peak height of reference solution
 V1- volume of sample solution mL
 V2- volume of standard solution mL
 m1- mass of sugar used in standard solution g
 m0- sample weight g

Volume of sample solution and volume of standard solution was kept same. $V1=V2$ $m1= 5\text{g}$ for fructose and 1.5g for glucose

$m0= 5\text{g}$

Four samples of turmeric powder and four samples of chilli powder were subjected to qualitative analysis and chromatographic analysis.

Turmeric Powder

Presence of yellow lead salts: Three grams of turmeric powder from each sample were placed in a test tube. Conc. It was then mixed with hydrochloric acid. Lead oxide yellow coloration was represented by magenta colouring.

Presence of chalk: In a test tube, 3g of the turmeric powder from each sample was added. It was then given a few drops of water and some hydrochloric acid. Chalk is present when there is effervescence.

Presence of aniline dyes: A few drops of water were added to each sample of turmeric powder. 3 ml of spirit were added to these. Aniline was present since the yellow colour immediately vanished.

Chromatographic Analysis

1gm of different turmeric samples (purchased locally) were dissolved in 20ml of solvent (Chloroform). The mixture was shaken for about 15 hours in a rotatory shaker. The mixture was then filtered using filter paper. The filtrate was used for the chromatography. Similar procedure was used for preparation of different turmeric samples. Next, a standard solution was prepared by dissolving 2mg of metanil yellow in 20ml of chloroform. This stock solution was diluted by dilution factor of 1:10 with the solvent and the diluted solution was used as a reference sample. The spots from the prepared standard solution of metanil yellow and from the extracted sample were placed on the TLC plate. The plate was placed in a jar containing mobile phase (mixture of n-hexane and ethyl acetate in the ratio of 9:1) for 1-1.5 hours. After this, the distance travelled by the spots were measured for samples and standard solutions along with the distance travelled by the solvent. The corresponding R_f values were calculated for each sample and standard solution sample by the formula given as $R_f = \frac{\text{Distance travelled by the spot}}{\text{Distance travelled by the solvent}}$.

Chilli Powder

Presence of red lead salts: Each sample of chilli powder received a small amount of diluted nitric acid. The remedy underwent filtering. Potassium Iodide was then added in two drops to the filtrate. The presence of red lead salts was suggested by the precipitate's yellow coloration.

Presence of oil soluble coal tar: Chilli powder samples weighing 3g each were placed in test tubes. The test tubes were well shaken after a few ml of ether solvent were introduced. A test tube holding 2 ml of diluted hydrochloric acid and an ether layer was decanted into. It was appropriately shook. The lower acid layer's distinct pink to red colour suggested the presence of oil-soluble coal tar.

Presence of brick powder: A beaker of water was added along with 3 samples of chilli powder. Pure chilli powder floated while brick powder settled.

Presence of Rodamine B: Chilli powder samples weighing 3g each were placed in a test tube. Acetone (5 ml) was added. Rodamine B was immediately visible as a red colour.

Chromatographic analysis

1gm of different chilli samples (purchased locally) were dissolved in 20ml of solvent (Chloroform) The mixture was shaken for about 15 hours in a rotatory shaker. The mixture was then filtered using filter paper. The filtrate was used for the chromatography. Similar procedure was used for preparation of different chilli samples. Next, a standard solution was prepared by dissolving 2mg of Sudan dye in 20ml of chloroform. This stock solution was diluted by dilution factor of 1:10 with the solvent and the diluted solution was used as a reference sample. The spots from the prepared standard solution of Sudan Red dye and from the extracted sample were placed on the TLC plate. The plate was placed in a jar containing mobile phase (mixture of n-hexane and ethyl acetate in the ratio of 9:1) for 1-1.5 hours. After this, the distance travelled

by the spots were measured for samples and standard solutions along with the distance travelled by the solvent. The corresponding Rf values were calculated for each sample and standard solution sample by the formula given as $R_f = \text{Distance travelled by the spot} / \text{Distance travelled by the solvent}$.

RESULTS

The results pertaining to the present investigation entitled “Detection and Quantification of Adulterants in Food items purchased from local stores in Kashmir Valley of Jammu and Kashmir” are presented in this chapter under the following headings

- Adulterants in Honey
- Adulterants in spices

Adulterants in Honey

Determination of Moisture content

The refractive index for different samples is presented in table 1. Data showed that honey purchased from the local market were not in standard ranges as per FSSAI standards. The Moisture content percentage for locally purchased honey was 21.2% and 22% respectively and the percentage for the honey purchased from the bee keeper was found to be 16%. While as per the FSSAI standards, the moisture content% should be <20.

Table 1: Moisture Analysis for honey

Sample	Refractive index (@20 deg)	%Moisture content (obtained from Wedmore data)	FSSAI Standard (%)
From local market	1.483	21.2	<20
From local market	1.486	22	<20
From Bee keeper	1.495	16.4	<20

Determination of HMF

The results obtained in the HMF level determination by spectrophotometer are presented in Table 2. It was found that 66.6% of the samples tested in this study contained the HMF level which exceeded 80mg/kg (FSSAI Standard). The samples which were purchased from the local market showed HMF value of 249.5mg/kg and 134.73mg/kg while the honey purchased from the bee keeper showed a value of 14.97mg/kg.

Table 2: HMF Analysis

C	A284	A336	HMF (mg/100 ghoney)	HMF (mg/kg honey)	FSSAI Standard (mg/kg)
From local market	0.4	0.2	14.97	249.5	<80
From local market	0.49	0.31	13.473	134.73	<80
From Bee Keeper	0.12	0.1	1.497	14.97	<80

Determination of fructose to glucose ratio

The results obtained with respect to present study on fructose to glucose ratio are presented in table 3. From the F/G analysis it was found that 2 out of three samples had high F/G ratios and didn't comply with FSSAI standards. The sample purchased from bee keeper had F/G value of 1.35 while the locally purchased market samples had a value of 1.55.

Table 3: F/G Analysis for Honey

Sample	A1	A2(F)	A2(G)	W(F) (g/100g)	W(G) (g/100g)	F/G	FSSAI Standard (F/G)
From local market	1.2	3	1.4	50	32.14	1.55	0.95-1.5
From local market	1.36	3	1.4	56.66	36.42	1.55	0.95-1.5
From Bee Keeper	0.95	3	1.4	39.58	29.31	1.35	0.95-1.5

Adulterants in spice

Turmeric Powder

The observations based on chemical tests are presented in table 4 It was found that;

1. Sample 1 contain Chalk
2. Sample 2,3 and 4 contain Aniline dyes
3. No presence of Yellow lead salts was found

Chromatographic Analysis

The data presented in Table 5 revealed that two out of four samples, had high Rf value than the standard value of Metanil Yellow. Sample 3&4 showed a Rf value 0.68 and 0.7 respectively while the standard Rfvalue should be 0.66. It was clear from the results that most of the samples were found adulterated with aniline dyes and metanil yellow.

Table 4: Observations based on Chemical Tests

SampleNo	Types of Adulteration		
	Yellow lead salts	Chalk	Aniline dyes
1	Not Present	Not Present	Not Present
2	Not Present	Not Present	Not Present
3	Not Present	Not Present	Present
4	Not Present	Not Present	Not Present

Table 5: Chromatographic Analysis for Turmeric

Sample no	Distance Travelled by solvent (cm)	Distance Travelled by sample (cm)	Rf value
Standard (Metanil yellow)	5	3.3	0.66
T1	5	2.6	0.52
T2	5	2.6	0.52
T3	5	3.4	0.68
T4	5	3.5	0.7

Chilli Powder

The data regarding the chemical tests is presented in table 6. It was found that sample 1, 3 and 4 contain Brick powder. No presence of Read lead salts, Oil soluble coal tar and Rodamine B was found.

Chromatographic Analysis

The results obtained with respect to Chromatographic Analysis are presented in table 7. The data revealed that none of the samples contain Sudan dyes the samples showed Rf value Of 0.63, 0.6,0.63 and 0.63 respectively. It is clear from the results that most of the samples were found adulterated with brick powder.

Table 6: Observations based on Chemical Tests

Sample.no	TYPES OF ADULTERATION			
	Red Lead salts	Oil soluble Coal Tar	Brick Powder	Rodamine B
1	Not Present	Not Present	Present	Not Present
2	Not Present	Not Present	Present	Not Present
3	Not Present	Not Present	Present	Not Present
4	Not Present	Not Present	Not Present	Not Present

Table 7: Chromatographic analysis for Turmeric

Sampleno	Distance travelled by solvent (cm)	Distance travelled by sample (cm)	Rf value
Standard (Sudandye)	5.5	4.5	0.81
C1	5.5	3.5	0.63
C2	5.5	3.3	0.6
C3	5.5	3.5	0.63
C4	5.5	3.5	0.63

DISCUSSION AND CONCLUSION

The items selected for the study were chosen which form the most common consumed and basic food products in households. These selected products included spices and honey. The qualitative and quantitative tests were conducted to determine the presence of different adulterants in these food products. The tests were done for the locally available products and from the origin supplier. The following conclusions were made for the different products:

For Spices, 4 samples of turmeric powder were tested for aniline dye, chalk powder, yellow lead salts and metanil yellow and it was found that three out of four samples i.e., 75% of the samples were adulterated with aniline dyes, one out of four sample was found to be adulterated with chalk powder and none of the sample showed the presence of yellow lead salts. Also, two out of four samples were found to be adulterated with Metanil yellow based on the Rf values obtained from HPLC. For Chilli Powder, four samples were tested for adulterants namely red lead salts, oil soluble coal tar, brick powder, Sudan dye and Rodamine B. The tests showed that none of the samples had red lead salts, oil soluble coal tar, Rodamine B and Sudan dye. While 75% of samples showed the presence of brick powder. Hence it was concluded that brick powder was the most common adulterant in chilli powder. Such colours and dyes should be checked and banned also so that they may not be used for enhancing the colour of spices and human health may be protected from the threats of these dyes and their cancerous effect.

In overall, it was concluded that all of the basic food items were found to be adulterated with atleast one adulterant. The adulteration as concluded from the study has been done preferably to gain the profit margins in the market. Thus, it is recommended that the Govt. should take precautionary steps and tight hold over the testing bodies to ensure an adulteration free product in market. This will be an aiding step for achieving "Suvast Bharat Mission" of Govt. of India.

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