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Research Article

Economically-motivated Adulterants in Milk and Infant Formulas Marketed in Sokoto Metropolis

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ABSTRACT

Milk adulteration is becoming a serious problem worldwide, particularly in underdeveloped and developing countries. The issue is attributed to factors such as low purchasing power, lack of suitable rapid test kits, degraded societal morals, inadequate monitoring, and lack of proper law enforcement. Repeated consumption of adulterated milk is likely to cause serious public health problems and raises significant concerns to the food industry and authorities. A total of fifteen (15) milk samples of different brands were randomly selected and purchased from local markets and supermarkets within Sokoto metropolis, Sokoto State, Nigeria. Twelve different adulterants (Sugar, starch, glucose, formalin, detergents, sodium chloride, hydrogen peroxide, pulverized soap, benzoic acid, nitrate, azo dye, and annatto) were tested qualitatively using standard analytical procedures. The findings from this study indicate that sugar and starch were detected in 60% and 27% of the milk samples, respectively. There was a 20% detection of each of hydrogen peroxide and detergents among the milk samples analyzed. Glucose, sodium chloride, and formalin were present in all the milk samples. However, pulverized soap, nitrate, benzoic acid, and colorants (azo dye and annatto) were not detected in any of the milk samples. The percentage values indicate the extent of specific adulteration of the milk samples by different adulterants irrespective of the amount (trace, moderate, and high). This study confirmed evidence of milk adulteration sold within Sokoto metropolis. Hence, stricter measures on control, registration, regulation, and awareness creation must be put in place to curb this growing problem.

Keywords: Adulterants; Adulteration; Health implications; Infant formulas; Milk

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INTRODUCTION

Dairy products in the form of either raw milk or lacteous derivatives are increasingly produced and consumed worldwide due to high nutritional contents, contributing to total daily energy expenditure (Handford *et al.*, 2015; Ceniti *et al.*, 2023). Recently, milk consumption has increased globally and it is estimated that world milk production will grow by 1.7% each year by 2028 (Lijuan, 2024). Milk is recognized as affordable and rich source of high quality protein, fat, carbohydrates, vitamins and essential minerals, playing a crucial role in the growth and development of infant

and children and necessary for maintenance of good health in adults (Ayub *et al.*, 2007; Ayza and Yilma, 2014; Windarsih *et al.*, 2021). Minerals like Na, K, Ca, and Mg are among the essential minerals required for a good nutrition and play an important role in normal body functions (Windarsih *et al.*, 2021; Rabiu and Abubakar, 2024). Due its importance, milk is traded worldwide; hence, many producers tried to keep it fresh and maintain its quality. However, unethical procedures might be employed to maximize profit and minimize economic losses during its transportation and marketing (Chauhan *et al.*, 2019; Anisha *et al.*, 2020). Milk adulteration is becoming a serious threat and a global concern to relevant authorities and general public, especially in developing countries where screening facilities are scarce (Spink and Moyer, 2011). Lack of awareness regarding food maintenance and safety, demand and supply gap and low purchasing capability of customer also contributed to this menace (Reddy *et al.*, 2017). The adulterated milk is invariably in circulation and will continue to pose a serious health risk to public health.

Studies have shown that milk was rated the second most adulterated food worldwide (Moore et al. 2012). Water and thickening agents such as starch, flour, glucose, urea, salt, chlorine, formalin, ammonium sulphate as well as preservatives like sodium bicarbonate, sodium carbonate, sodium hydroxide and calcium hydroxide are common adulterants found in milk and milk products. Some toxic substances had been reported in milk (Reddy et al., 2017; Yadav et al., 2023). The adulteration of Chinese-manufactured infant milk formula with melamine in 2008 sent shock waves worldwide as thousands of infants were hospitalized, and significant fatalities were recorded following the consumption of melamine-tainted milk (Gawali, 2022). Adding melamine would allow milk dilution (increased quantity and financial gain) while keeping the nitrogen content of milk within the established limit as determined by the conventional Kjedahl method (Cheng et al., 2010; Reddy et al., 2017; Windarsih et al., 2021). Added urea usually provides whiteness and increase non-protein nitrogen content of the milk. Sucrose is added, just to increase the carbohydrate content and density of the milk. Starch is added to increase solid-notfat (SNF) content in the milk (Chauhan et al., 2019). Generally, neutralizers are added in synthetic milk to neutralize the acidic effect. The toxic hydrogen peroxide (H₂O₂) is added to milk to prolong its freshness. While some added detergents help to emulsify and dissolve the oil in the milk and this gives a typical characteristic white colour of milk (Singuluri and Sukumaran, 2014). These added substances might equally facilitate growth and development of some microorganisms. Hence, the addition of some preservatives come into play. Boric acid, formalin, sodium carbonate (Na₂CO₃), sodium bicarbonate (NaHCO₃), salicylic acid, benzoic acid and sodium azides are usually added to preserve the milk for longer period of time (Del Olmo et al., 2017; Windarsih et al., 2021; Yang et al., 2022).

As Africa and other developing continents are still grappling with low milk production to cater for their booming population, adulteration of dairy products will most likely persist in the absence of adequate regulatory control and deterrence. Therefore, presence of these adulterants which are quite different from those declared in labelled milk and its products is a matter that requires prompt actions from all stakeholders including consumers, producers, and regulatory agencies.

Given the dire consequences of human consumption of these adulterants, which often result in toxicities and allergies, and in some cases could be fatal, it is then imperative to revisit and chart a path towards the identification and cataloguing of existing economicallymotivated adulterants (EMA) practices in the dairy products supply chain in Nigeria. Therefore, the present study aimed at determining the economicallymotivated adulterants added in milk.

MATERIALS AND METHODS

Different analytical chemicals and dyes were used, which included resorcinol, Barfoed's reagent, silver nitrate, ferric chloride, concentrated hydrochloric acid, diphenylamine sulphate, mercuric chloride, concentrated sulphuric acid, methylene blue dye, phenolphthalein indicator, sodium bicarbonate and turmeric paper. All these chemicals and reagents used were of analytical grades.

Study Area

The study was conducted within the Sokoto metropolis, Sokoto State, Northwest Nigeria.

Collection of Samples

A total of forty-five (45) commercial milk samples, including infant formulas, liquid milk and milk powders, from fifteen different brands were randomly selected and purchased from local markets and supermarkets within Sokoto metropolis, Sokoto State, Nigeria. The samples were analyzed in the Department of Biochemistry and Molecular Biology, Usmanu Danfodiyo University Sokoto. Efforts were made to acquire milk samples not listed in the National Agency for Food and drug administration and Control (NAFDAC) dairy products database.

Test for adulterants

The milk samples were tested for the following adulterants: sugar, starch, glucose, formalin, detergents, sodium chloride, hydrogen peroxide, pulverized soap, benzoic acid, azo dye and annatto

Test for sugar

About 5 mL of milk sample was taken into a clean test tubes and 1 mL of concentrated HCl was gently added, followed by addition of 0.1g of resorcinol. The mixture was heated in water bath for about 5 minutes. The mixture that turned red in color indicated the presence of added sugar (Sharma *et al.*, 2012; Kamthania *et al.*, 2014).

Test for starch

In well labelled test tubes, 3 mL of each milk sample was taken into respective test tubes and boiled in a water

bath for 5 minutes. The solution was allowed to cool and 1% iodine solution was then added and mixed thoroughly. The mixture observed with black colour confirmed the presence of added starch (Sharma et al., 2012; Arvind Singh et al., 2012).

Test for glucose

One milliliter (1 mL) of milk sample was dropped into a clean test tube, 1 mL of Barfoed's reagent was added and the mixture was heated for at least 3 minutes in a boiling water bath. The test tube was allowed to cool under running tap water. The mixture that developed deep blue colour confirmed the presence of added glucose (Sharma et al., 2011).

Test for benzoic acid

Into a clean test tube, 5 mL of milk sample was added and acidified with sulfuric acid, about 0.5% FeCl₃ solution was then added drop by drop and mixed thoroughly. Sample solution with buff colour confirms the presence of added benzoic acid

Test for hydrogen peroxide

About 5 ml of milk sample was taken into a test tube, an equal volume of raw milk and 4-5 drops of a 2% solution paraphenylenediamine were of added. The development of a blue colour confirmed the presence of added hydrogen peroxide (Arvind Singh et al., 2012; Kamthania et al., 2014).

Test for sodium chloride

Two milliliters (2 mL) of milk sample was taken into a clean test tube, 0.1 mL of 5% potassium chromate and 2 mL of 0.1 N silver nitrate were added accordingly. Appearance of Yellow precipitate in the solution confirmed the presence of added sodium chloride (Sharma et al., 2011)

Test for nitrate

About 10 mL of milk sample was taken into a clean beaker, 10 mL of mercuric chloride solution was added and mixed thoroughly. The solution was filtered using Whitman paper No. 42. One milliliter (1 mL) of filtrate was transferred to a separate test tube and 4 mL of diphenyl benzidine reagent was added. The mixture that developed blue colour confirmed the presence of added nitrate (Sharma et al., 2011).

Test for pulverized soap

Ten milliliters (10 mL) of milk sample was taken into a clean test tube, an equal volume of hot water was added and about 2 drops of phenolphthalein indicator were the added to the solution. The development of a blue colour confirmed the presence of added soap (Arvind Singh et al., 2012; Kamthania et al., 2014).

Test for detergent

About 2.5 mL of milk sample was taken into a 10 mL clean test tube, 0.5 mL of Methylene blue dye solution was added to it and 1 mL chloroform was added and vortex for about 15 seconds. The mixture was centrifuged at 1100 rpm for exactly 3 minutes. Appearance of intense blue colour in lower layer of the test tube confirmed the presence of added detergent (Arvind Singh et al., 2012).

The test for coluring matter

A 10 mL milk solution was made alkaline using sodium bicarbonate, and a strip of filter paper was dipped for two hours. Appearance of red colour on the filter paper confirmed the presence of added annatto dye (Lechner and Klostermeyer, 1981).

A few drops of hydrochloric acid were added to the milk sample solution. Appearance of pink colour confirmed the presence of added azo dyes (DE Souza et al., 2000).

Test for formalin

In a 500 mL volumetric flask, 1 mL of 10 % FeCl₃ solution was added, and concentrated hydrochloric acid was used to make up the volume. From the solution, 5 mL was taken and transferred into a test tube containing 5 mL of milk sample. The mixture was kept for about 3-4 minutes in a boiling water bath. Appearance of brownish pink colour confirmed the presence of added formalin (Sharma et al., 2012).

RESULTS

The 45 samples of different types of powdered milk (including infant formulas) showed different characteristics (Table 1).

The results of tested adulterants in the milk samples presented in Table 2, indicated that glucose and formalin were detected (100% respectively) in all the milk samples analyzed. Sugar (60%), starch (26.7%), sodium chloride (26.7%) and detergent (20%) were also detected in some milk samples. However, pulverized soap, colourants, nitrate and benzoic acid were not detected (0%) in any of the analyzed milk samples.

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S/No	Sample	Number Examined	Class	Protein source	Target Age Group 0-6		
1	А	3	Enriched formula	Milk			
2	В	3	Enriched formula	Milk	6-12		
3	С	3	Enriched formula	Milk	0-6		
4	D	3	Enriched formula	Milk	0-12		
5	E	3	Enriched formula	Milk	0-12		
6	F	3	Cereal-Mixed formula	Wheat Based	6 – 36		
7	G	3	Cereal-Mixed formula	Wheat Based	6-12		
8	н	3	Cereal-Mixed formula	Wheat Based	6 - 12		
9	I	3	Enriched formula	Milk	0-6		
10	J	3	Enriched formula	Milk	6 - 12		
11	К	3	Soya based formula*	Soy	>36		
12	L	3	Soya based formula*	Soy	>36		
13	М	3	Soya based formula*	Soy	>36		
14	Ν	3	Soya based formula*	Soy	>36		
15	0	3	Soya based formula*	Soy	>36		

*Not registered by National Agency for Food and Drugs Administration and Control (NAFDAC)

Table 2: Occurrence of different adulterants in the milk samples
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S/No	Adulterants	Α	В	С	D	Ε	F	G	н	I.	J	К	L	Μ	Ν	0	% of Occurrence
1	Sugar	-	-	-	-	-	+	+	-	+	+	+	+	+	+	+	60
2	Starch	-	-	-	-	-	+	+	+	-	-	-	-	+	-	-	26.7
3	Glucose	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	100
4	NaCl	++	-	-	-	-	-	-	-	-	-	-	-	++	++	++	26.7
5	H ₂ O ₂	-	-	-	-	-	+	+	+	-	-	-	-	-	-	-	20
6	Formalin	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	100
7	Detergent	-	-	-	-	-	-	-	+	-	-	-	-	+	-	+	20
8	Pulverized soap	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0
9	Azodye	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0
10	Annatto	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0
11	Nitrate	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0
12	Benzoic acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0

DISCUSSION

Milk is agreeably consumed worldwide and considered second most commonly adulterated food (Azad and Ahmed, 2016; lonescu *et al.*, 2023). Adulteration of milk is becoming a serious global problem. The increase and fraudulent activity of milk adulteration has raised an alarming concern to authorities, consumers and manufacturing industries. Hence, the general public often become victim of diseases associated with consuming adulterated milk (Swar *et al.*, 2021). The adulterants found mostly in milk include those that do not have serious health risk (water, vegetable proteins and whey) and those with likely potentials of causing health risk like formalin, benzoic acid, detergents, chlorine and ammonium sulphate (Gawali, 2020; lonescu *et al.*, 2023).

Our study observed that 60% of the analyzed milk samples contain added sugar. Chugh and Kaur (2021) reported 9% of the milk samples analyzed contain added sugar. Moreover, about 22% of milk samples analyzed by Singulurin and Sukumaran (2015) were adulterated with sugar. Table sugar like sucrose is commonly added to the milk just to increase the carbohydrate, the solids- not-fat (SNF) content of milk and the density of milk. Therefore, presence of sugar in the milk may indicate that it was used as sweetener, therefore mimicking the natural sweetness of milk (Singulurin and Sukumaran, 2015). The findings of this study also indicated that 27% of the analyzed milk samples were adulterated with starch particularly wheat and soy-based milk formulas. Starch usually increases SNF contents of the milk. The added starch has no impact on colour and taste. Excessive starch content in milk is reported to cause diarrhoea due to its indigestibility in colon and may be dangerous for diabetes patients (Singuluri et al., 2014; Gawali, 2020; Visciano and Schirone, 2021).

The added NaCl increase the density of milk when adulterated with water. All the samples analyzed in this study had excess of chloride, which will likely distort acid

base balance of blood and can cause clogging in arteries which may consequently lead to heart related problems (Gawali, 2020). Surprisingly, all the samples analyzed (100%) in this contain sodium chloride against the 18% of samples reported by Singulurin and Sukumaran (2015). Hydrogen peroxide is commonly used in milk and milk products as preservative to keep the milk fresh for longer period time. It is non-selective germicide and even more active on lactic bacteria than pathogenic microflora (Momtaz et al., 2023). About 20% of the analyzed samples particularly wheat-based formulas contained significant amount of hydrogen peroxide. A similar study conducted in India reported the presence of hydrogen peroxide in 32% of the analyzed milk samples (Singulurin and Sukumaran, 2015). The addition of hydrogen peroxide in milk, even in small quantities, is reported to induced oxidation of milk fat, cause damage to gastrointestinal cells and eventually leads to gastritis and inflammation of the intestine (Gawali, 2020; Ivanova et al., 2019). Formalin is used as preservative to maintain the freshness of milk over long period of time during transportation and storage. Unfortunately formalin is a toxic substance and has long been considered as carcinogenic, causing liver and kidney damages (Azad and Ahmed, 2016; Mabood et al., 2017). Our study revealed that all the samples analyzed were adulterated with formalin. Thus, persistent consumption of such a milk brand could be a potential risk factor for carcinogenesis. Singulurin and Sukumaran (2015) reported that 32% of the milk samples analyzed were adulterated with formalin.

The results of the present study also confirmed the presence of detergent in some milk samples; about 20% of the samples were identified to contain added detergents. Singulurin and Sukumaran (2015) reported 44% of the milk samples analyzed were adulterated with detergents. These substances are added to milk to improve the cosmetic nature of milk and make it thicker (Poonia *et al.*, 2019; Avula *et al.*, 2022). However, detergents are known to cause gastrointestinal disturbances and kidney malfunction (Chauhan *et al.*, 2019; Gawali, 2020).

The detection of detergent in some of the milk samples observed in the present study could be one of the contributing factors for the high incidence of kidney failure in Nigerian children (Ibrahim *et al.*, 2025).

CONCLUSION

Several chemical and natural substances are added in milk mostly to maximize financial benefits. Adulterated milk and milk products are known to pose serious health risk to public especially children and elderly. In this study, sugar, starch, glucose, sodium chloride, hydrogen peroxide, detergents and formalin were detected in some milk samples, especially wheat-based formulas and not registered by National Agency for Food and Drug Administration and Control (NAFDAC). In fact, these added substances compromised the quality of milk and need to be monitored regularly to protect the health of the general public. Therefore, it becomes imperative that the academic communities, relevant regulatory agencies, and the general public come together and fight this menace through an efficient and reliable quality control system, regular monitoring, continuous research, awareness, and provision of simple and affordable adulteration rapid detection kits. The Nigerian regulatory laws on foods and food products must be strictly implemented and enforced. The regulatory agencies must be fully equipped with manpower, equipment and regular training to acquaint themselves with the state-of-the-art facilities in the sector. Rapid, reliable and inexpensive test kits should be made available even at local communities to detect various toxic adulterants. Offenders should be severely punished; fines and jail terms will help reduce adulteration of food substances. Punishment should also be served to enforcement personnel who allowed or permitted the registration, production, distribution, and marketing of adulterated food.

Declaration of Competing Interest

The authors declared no known competing interests or personal relationships that could have appeared to influence the work reported in this paper

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