

Original article

## Determination of Carboxymethyl Cellulose, Total Dissolved Solids, pH, and Microbiological Quality of Ice Cream Samples Collected from Libyan Markets

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### ABSTRACT

#### Keywords:

Ice Cream, CMC, pH, Tds, Microbial.

Ice cream is one of the most food consumer during different seasons, it contains different chemical compounds and food additives. This study aimed to estimate the contents of Carboxyl Methyl Cellulose (CMC) in many ice cream which found in the major supermarkets in some Libyan cities. Also, the pH and total dissolved salts. This study aims to measure the levels of Carboxyl Methyl Cellulose (CMC) in various ice cream samples collected from the local markets (Libya), where twenty-one types were purchased from different supermarkets, including imported and local ice cream samples. The spectrophotometric methods were used in this study for carboxyl methyl cellulose, while the total dissolved solids (TDS) and PH values were estimated by PH mater, including a sensor for TDS values. The concentrations of CMC of the ice cream samples fluctuated in the range of (207.33 – 1250.66 ppm ), the high contents of ( 1250.66 ppm ) were recorded in the samples of (Ice Queen, Jordan), whereas the low contents were recorded in the samples of (SNICKERS CRISP, France). The results recorded wide variations in the CMC of the studied samples. (Table 2 and Figure 4). The contents of TDS values ranged between (26.1 – 191 ppm), the high contents were recorded in the samples of (SNICKERS, Poland), whereas the lower values were observed in the samples of (AIMADHAKSTRA WBERRY, Libya). For the pH values, most of the samples gave acidic values (5.3 – 7.14). According to the results recorded in this study, there are wide variations in Carboxyl methyl cellulose in the studied samples of ice cream. The values of Total Dissolved solids /Salts showed wide variations, on the other side the pH values showed that most of the studied are lied in the acidic scale, which may be harmful to human health. The presence of CMC in the samples is very dangerous for the human consumer, where most of the recorded values are above 100 ppm, which is recommended by the WHO. For the microorganism study, the results recorded the presence of some bacteria and Fugal species in the selected samples; these organisms are associated with many diseases and have a negative effect on human health. Ice cream is one of the most consumed foods by children and others.

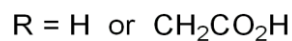
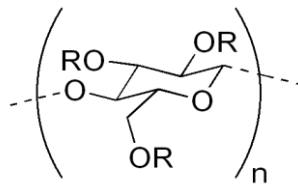
### Introduction

Ice cream is a very popular cold dessert. It is usually made by mixing milk or cream with sugar and flavors like fruit or vanilla. To make it smooth, the mixture is stirred a lot while it freezes. This stops ice from forming and puts air inside. It stays hard when it is very cold, but gets soft when it warms up a little. Different countries have different rules about what can be called "ice cream." In America, there must be a certain amount of cream. If it doesn't, the box must say "frozen dairy dessert." But in countries like Italy, all frozen treats use the same word. There are also many kinds made without cow's milk, using things like soy or coconut milk, so anyone can eat them. You can enjoy ice cream in a bowl, in a cone, or mixed into milkshakes and sundaes [1].

Ice cream might seem simple, but it is actually a carefully balanced scientific creation. It is made of water, fat, milk proteins, sugar, and air. Normally, fat and water don't mix, but proteins in the milk act as a bridge to hold them together. Whipping air into this mixture gives ice cream its light, fluffy texture. The sugar in the mix is also important because it lowers the freezing temperature of the water. This means not all the water freezes solid, which stops the ice cream from becoming too hard to eat. However, if there is too much natural milk sugar (lactose), it can make the texture weird or gritty. To make sure companies get this balance right, the U.S. government has strict rules. To be legally called "ice cream," it must have over 10% milk fat, specific amounts of milk solids and sweeteners, a tiny bit of stabilizers, and about 55% to 64% water [2]. The compositional standards of ice cream are delineated by weight rather than volume. Given that incorporation of air (overrun) can constitute up to 50% of the product's volume, weight-based

metrics provide a more accurate representation for dietary assessment. Consequently, even low-fat variants remain calorically dense due to elevated sugar content.

Regulatory frameworks vary by jurisdiction; in Canada, the Food and Drugs Act distinguish between "ice cream" and "ice cream mix." The finished product must contain a minimum of 10% milk fat and 180 g/L of total solids, with a reduced-fat threshold of 8% permitted for variegated flavors containing fruit or confections. The unfrozen mix requires a minimum of 36% solids and permits specific concentrations of stabilizers, pH adjusters, and emulsifiers. Structurally, ice cream is classified as a colloidal foam, the stability of which is heavily dependent on dietary emulsifiers such as polysorbate and soy lecithin. However, the physiological impact of these additives is subject to scrutiny; a 2015 murine study indicated that polysorbate 80 and carboxymethylcellulose might induce metabolic syndrome, obesity, and inflammatory bowel conditions [3&4]. Carboxymethyl cellulose (CMC), also designated as cellulose gum, is a cellulose derivative synthesized via the substitution of carboxymethyl groups (-CH<sub>2</sub>-COOH) for the hydroxyl groups on the glucopyranose monomers constituting the cellulose polymer backbone. The sodium salt form, sodium carboxymethyl cellulose, is the most widely utilized iteration in practical applications. Previously, this compound was commercially distributed under the registered trademark Tylose by SE Tylose [5,6].



**Figure 1. Carboxyl methyl cellulose (CMC).**

Carboxymethyl cellulose (CMC) is a highly versatile thickener and emulsion stabilizer used in a wide variety of food and non-food items. Its popularity stems from the fact that it is non-toxic, hypoallergenic, and derived from natural plant fibers like softwood pulp or cotton. Beyond the kitchen, CMC is a key ingredient in toothpaste, laxatives, diet pills, water-based paints, detergents, paper products, textile sizing, reusable heat packs, and even leather crafting. In the food world, it is listed as E466 (or E469 if enzymatically broken down) and is heavily relied upon to stabilize emulsions—such as in ice cream—and to improve the texture of gluten-free and low-fat foods. Interestingly, winemakers also use CMC to prevent tartrate crystals from forming in wine [6]. This keeps wine stable without refrigeration, potentially saving massive amounts of electricity in warmer climates by offering a more effective and longer-lasting alternative to traditional metatartaric acid [7].

In winemaking, CMC alters potassium bitartrate (KHT) crystals by causing them to lose two of their seven faces, resulting in a flatter shape. Because CMC carries a negative charge at wine pH levels, it binds to the positively charged potassium ions on the crystal's surface. This slows down crystal growth because the CMC and bitartrate ions essentially compete for the same binding spots. In the food industry, CMC powder has revolutionized ice cream making by allowing manufacturers to create ice cream without traditional churning or extremely cold salt-ice mixtures. In baking, CMC improves the quality of breads and cakes while lowering costs by reducing the required amount of fat. When used in biscuits, it acts as an emulsifier that evenly distributes fat throughout the dough [8]. This prevents sticking, ensuring the biscuits release cleanly from molds and cutters without losing their shape, while also cutting down on the need for egg yolks or additional fat. Furthermore, CMC helps evenly blend flavor oils into candies for a smoother texture and serves as an emulsifier in chewing gums, margarines, and peanut butter. Overall, ice cream remains one of the most popular foods consumed year-round, relying on a complex mix of chemical compounds and additives like CMC. This study aimed to estimate the contents of Carboxyl Methyl Cellulose (CMC) in many ice cream which found in the major supermarkets in some Libyan cities. Also, the pH and total dissolved salts were measured in the selected ice cream samples.

## Methods

### Sampling

Different ice cream samples were collected from markets in some Libyan markets, the samples including different types of Ice Cream produced from different countries. The samples were given in (Table 1).

**Table 1. The types of the studied samples**

Sample No	Sample type	Sample No	Sample type
1	Heart Strawberry	12	XL
2	SOrbeto Mango	13	GLACE VANILLA
3	CANDY BAR	14	MAGNUM AIMOND CONE
4	Anguria Mango	15	Häagen-Dazs
5	Anguria Watermelon	16	LONDON DAIRY VANILLA
6	First	17	Giles Chocolate
7	Ice Queen	18	BOM BOM
8	SNICKERS CRISP	19	Danette Selection
9	Lotus Biscoff	20	AIMADHAK STRAWBERRY
10	Giles vanilla	21	V.I.P
11	SNICKERS		

### Samples Preparation

All chemical reagents utilized in the experiments, specifically sodium carboxymethyl cellulose (CMC) and methyl blue (MB), were procured from Macklin Reagent Co. Ltd. (Shanghai, China) and Alfa Aesar (Massachusetts, USA), respectively. Deionized water served as the blank for all UV-Vis analyses. To prepare the CMC stock solution (1.013 g/L), 0.100 g of CMC powder was dissolved in a beaker of water and stirred until completely transparent. This solution was subsequently transferred to a 100-mL volumetric flask and diluted to the mark with deionized water. Similarly, a 20 mg/L MB stock solution was formulated by dissolving 20 mg of MB powder in deionized water within a 1000-mL volumetric flask. Spectroscopic measurements were conducted using a DU-800 UV/Vis spectrophotometer fitted with a 1 cm quartz cuvette.

### Determination of CMC

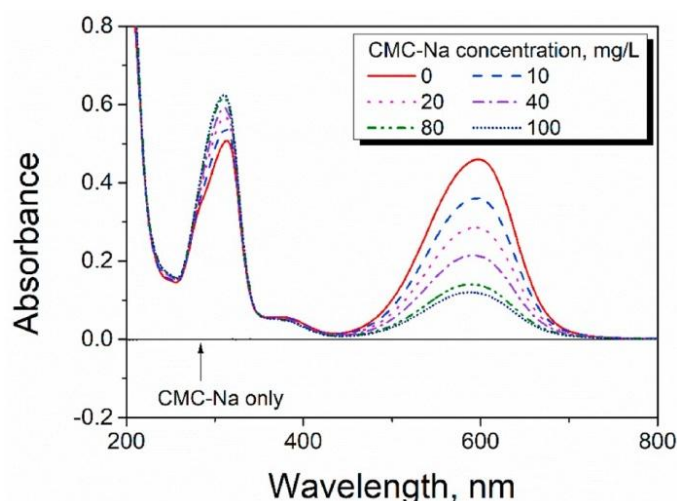
The CMC concentrations in the selected samples were quantified using the methyl blue method, given by previous studies [ ].

### Procedures in the measurement

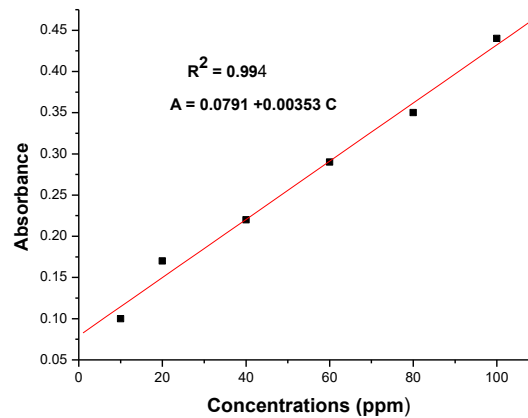
A mixture of 5 mL of CMC solution and 5 mL of MB stock solution was thoroughly combined in a 20-mL beaker. The UV-Vis spectrum of this solution was then analyzed to record the absorbance within the specified wavelength range.

### Method calibration

A calibration curve was constructed using standard solutions with CMC-Na concentrations of 10, 20, 40, 60, 80, and 100 mg/L, which were prepared by serially diluting the 1.013 g/L CMC stock solution. The absorbance of these standards at the target wavelength was recorded via spectroscopic measurement. Using this calibrated method, the CMC concentrations in the ice cream samples were specifically determined at a wavelength of 597 nm.



**Figure 2. Spectra of MB/CMC solutions system.**



**Figure 3. The standard Curve of CMC Solutions**

#### **Determination of pH**

The pH values were determined by pH meter (Type Toldo).

#### **Total Dissolving Solids (T.D.S).**

The total dissolved solids (TDS) were measured by a TDS meter (Type HANNA).

#### **Assay of bacteria and Fungi activities**

The bacteria and Fungi activities were carried out according to the agar method, where 0.5 g of the ice cream sample was dissolved in 50 ml of distilled water.

#### **Preparation of the sample for the test**

Approximately 2.5 g of the frozen product was aseptically added to 22.5 mL of buffered peptone water and shaken vigorously to form the master dilution. A serial dilution series was then performed by transferring 1 mL of the suspension into 9 mL of saline ( $10^{-1}$  dilution) using sterile pipettes, mixing thoroughly between each step. The dilutions were carried out sequentially up to  $10^{-6}$ , alongside a control tube.

#### **Test of (coli form test)**

We used a modified MacConkey broth containing Durham tubes to detect gas production. We prepared three sets of five tubes: the first set held 10 mL of double-strength broth, while the second and third sets held 5 mL of single-strength broth. We inoculated 10 mL of the sample into the first set, 1.0 mL into the second set, and 0.1 mL into the third set. After incubating the tubes at 37°C for 24 hours, we looked for positive results, which were indicated by acid production (color change) and gas trapped in the Durham tubes.

### **Results**

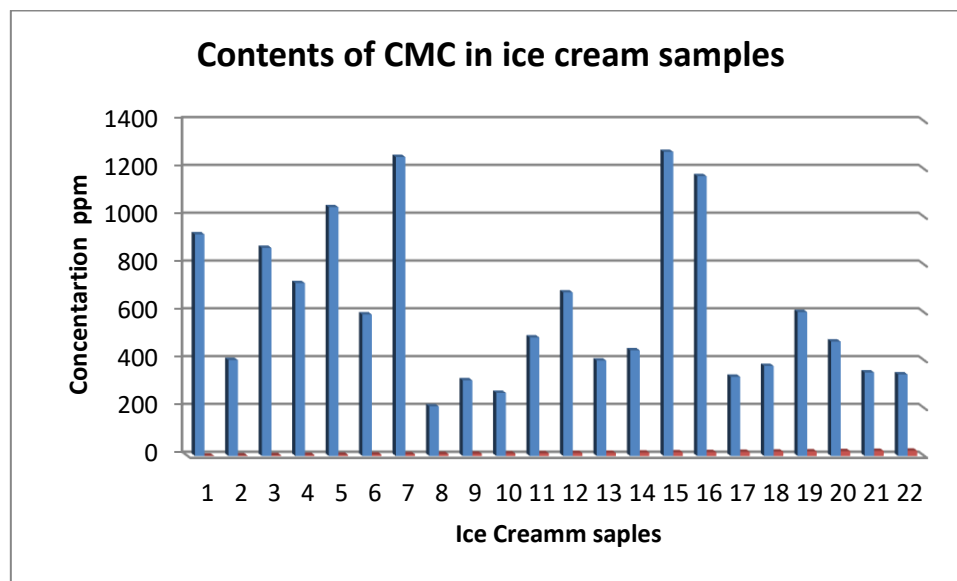
#### **Carboy methyl cellulose contents**

CMC levels in the ice cream samples showed significant variation, ranging from 207.33 ppm (SNICKERS CRISP, France) to 1250.66 ppm (Ice Queen, Jordan), as detailed in (Table 2) and Figure 4. TDS values also fluctuated widely between 26.1 mg/L (AIMADHAKSTRA STRAWBERRY, Libya) and 191 mg/L (SNICKERS, Poland). Additionally, most samples exhibited an acidic pH, with values falling between 5.3 and 7.14 (Table 2, Figures 4 & 5).

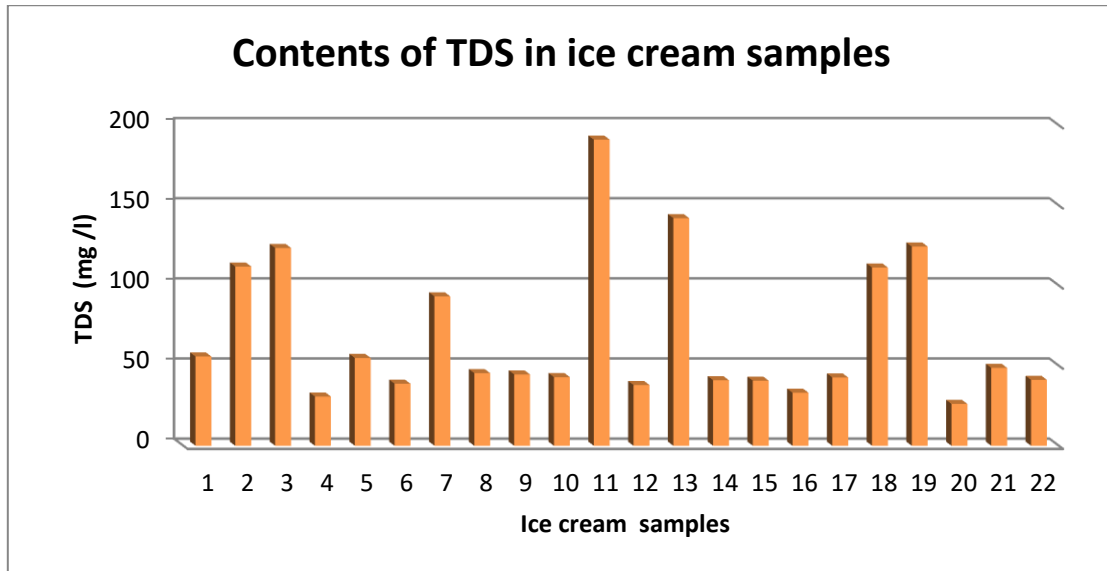
**Table 2. The contents of the CMC of the studied Ice Cream samples.**

No	sample	The manufacture company	Country	Concentrations CMC (ppm)	TDS (mg/l)	PH
1	Heart Strawberry	Ice Queen	Jordan	927	55.7	5.44
2	SORbeto Mango	Ice Queen	Jordan	401.33	111.8	7.10
3	CANDY BAR	Ice Queen	Jordan	870.5	123.4	7.14
4	Anguria Mango	Ice Queen	Jordan	723.16	30.7	6.30
5	AnguriaWatermelon	Ice Queen	Jordan	1040.83	54.8	6.29
6	First	Ice Queen	Jordan	591.83	38.7	5.92
7	Ice Queen	Ice Queen	Jordan	1250.66	93.2	6.30

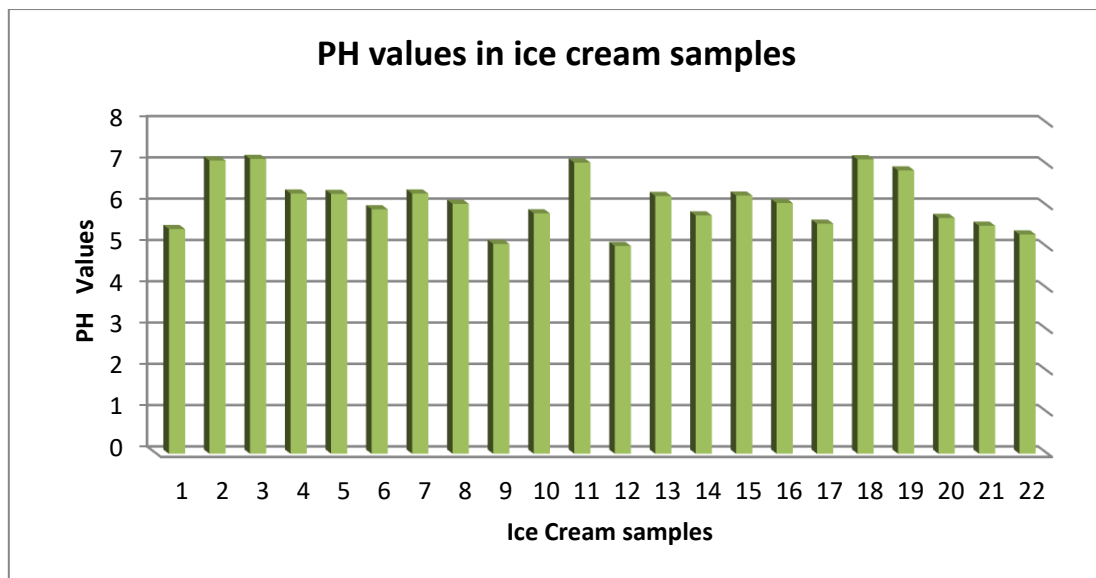
8	SNICKERS CRISP	SNICKERS	France	207.33	45.3	6.05
9	Lotus Biscoff	Lotus	Belgium	316.33	44.5	5.08
10	Giles vanilla	Al Naseem	Libya	263.83	42.8	5.82
11	SNICKERS	SNICKERS	Poland	495.66	191	7.05
12	XL	OLA	Tunisia	684	37.9	5.03
13	GLACE VANILLA	CÔTE D'OR	Belgium	399	142	6.24
14	MAGNUM ALMOND CONE	MAGNUM	Belgium	441.83	40.9	5.77
15	Häagen-Dazs	Bounty Brands	France	1272	40.6	6.25
16	LONDON DAIRY VANILLA	LONDON DAIRY	UAE	1170.33	33.1	6.07
17	Giles Chocolate	Al Naseem	Libya	331.16	42.6	5.57
18	BOM BOM	Ice Queen	Jordan	376.5	111.2	7.13
19	Danette Selection	Istanbul uretici	Turkey	602	124.3	6.86
20	AIMADHAK STRAWBERRY	AIMADHAK	Libya	478.66	26.1	5.71
21	V.I.P	Amro Company	Egypt	349	48.6	5.52
22	Giles Strawberry	Al Naseem	Libya	341.16	41.1	5.31



**Figure 4.** The contents of the CMC of the studied samples.



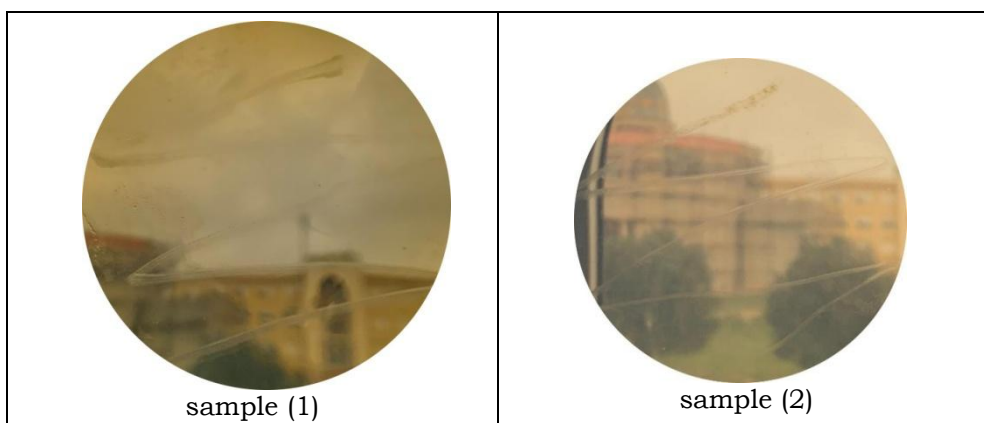
**Figure 5.** The contents of the TDS of the studied samples.

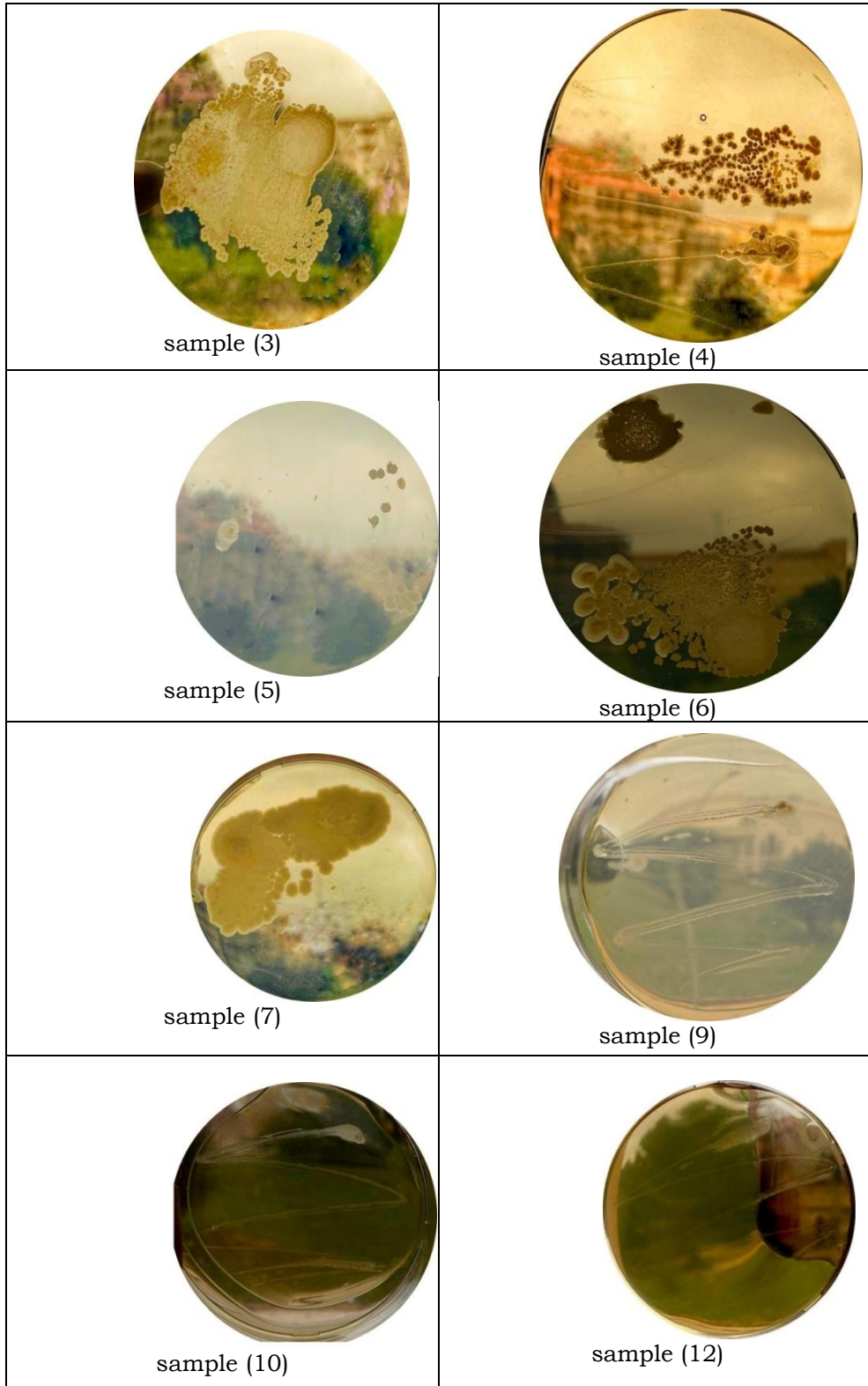


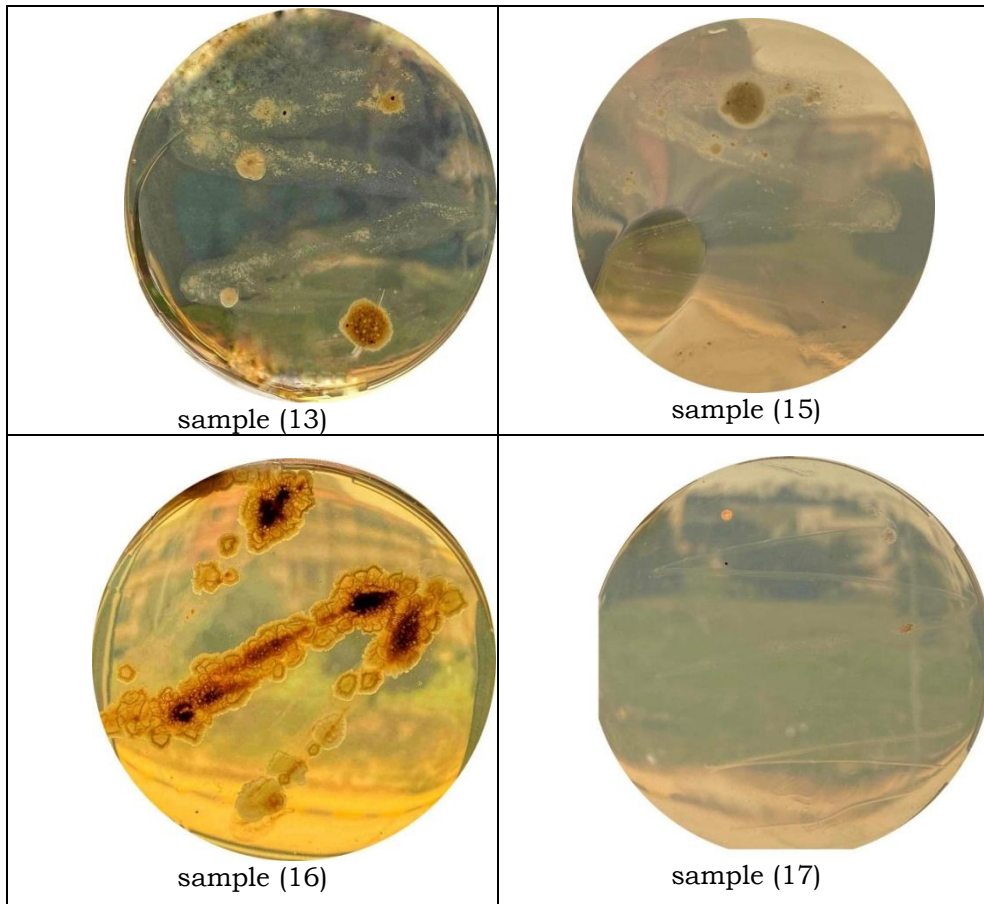
**Figure 6.** The pH of the studied sample

**Bacteria and Fungi activities**

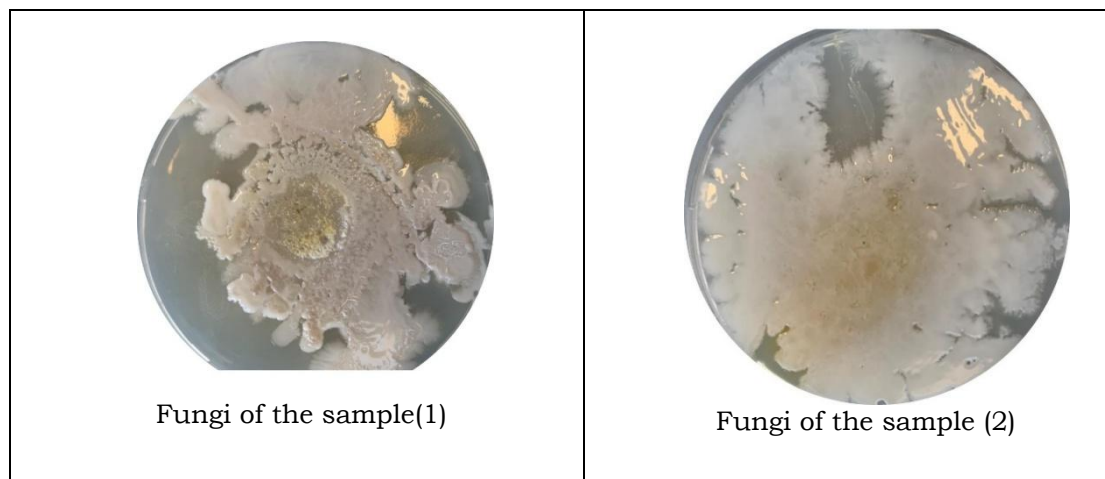
The results of the Bacteria and Fungi activities showed the presence of some types of bacteria and Fungi species in the studied samples. The results are shown in the following images.

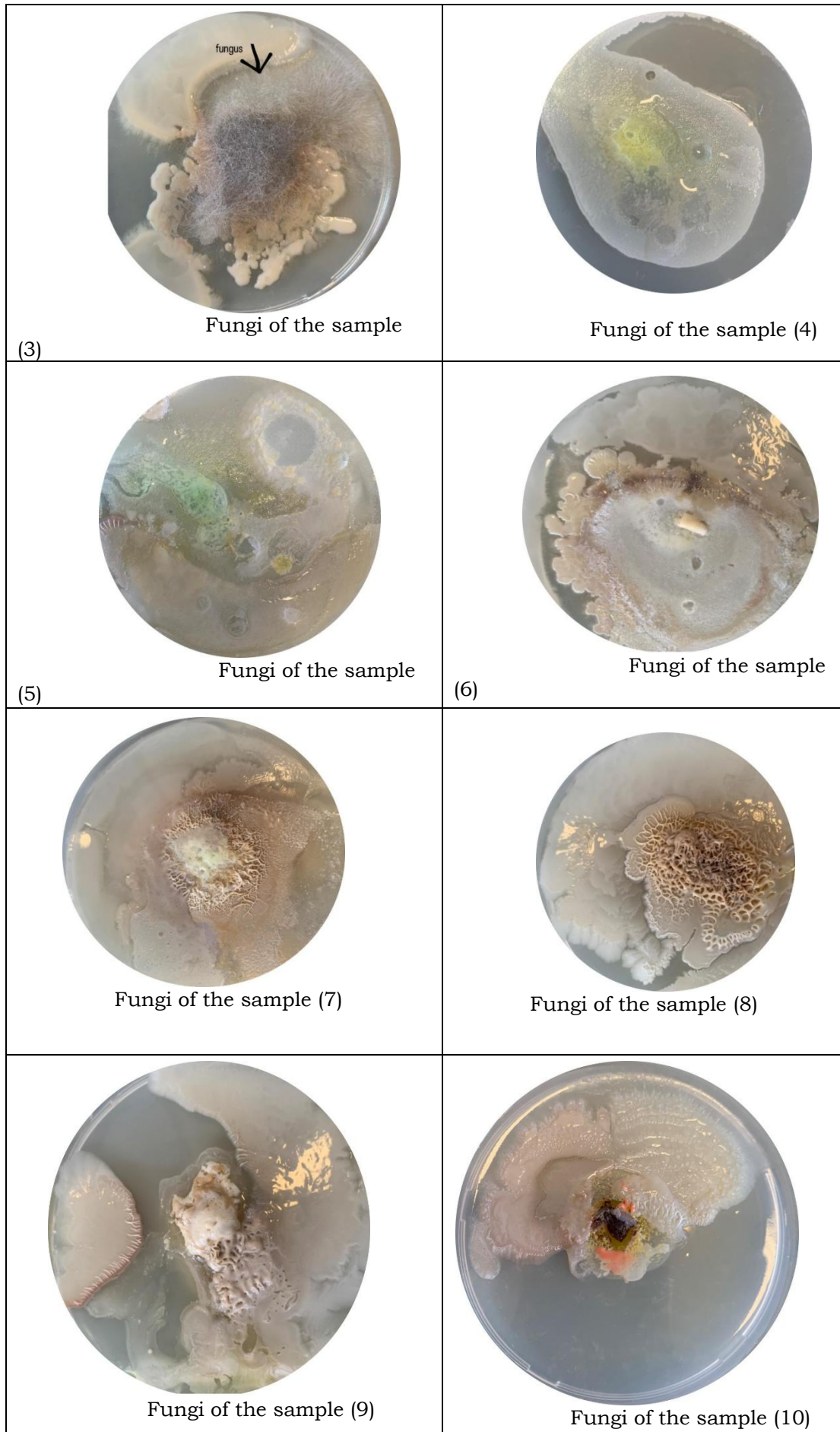


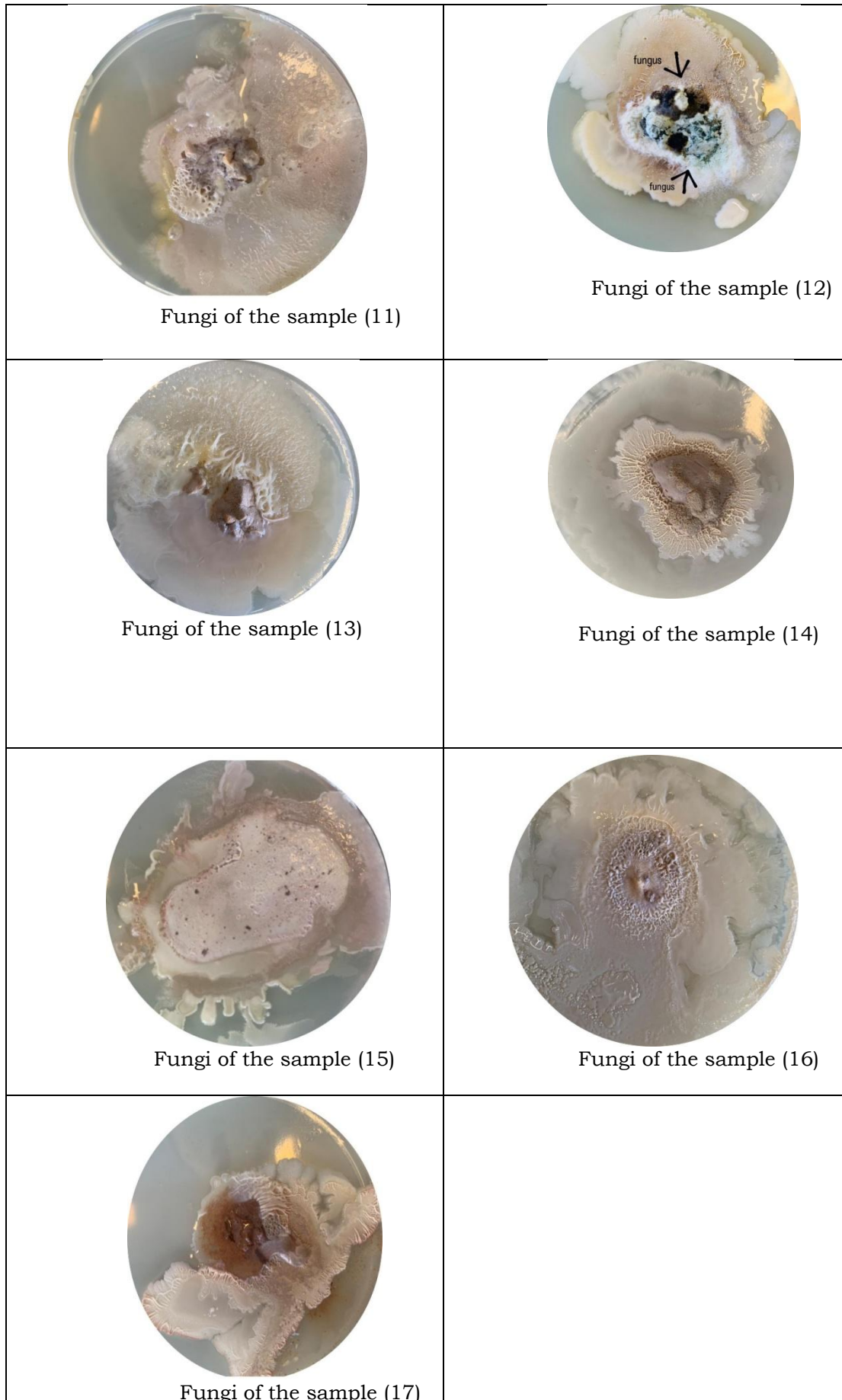




On the other side the anti -Fungi activities were shown in the following images.







## Discussion

The results of this study recorded the presence of carboxyl methyl cellulose (CMC) in most of the studied samples; the values are higher than the recommended values by the WHO. The CMC in one of the compounds added to ice cream, but higher values than 100 ppm caused many problems for human health. The ice cream contains milk. Milk is a nutritious basic food. It is rich in proteins, carbohydrates, fats, calcium, phosphorus, riboflavin, and other B vitamins. Milk and milk products are most popular and are widely consumed by people all over the world. There are several kinds of milk products available, and among them, the most important is ice cream. Ice cream is a frozen dairy Product. It is a rich source of milk nutrients and a luxury food product [9].

Ice cream is the result of an evolution covering a period of about five centuries. It may be said to have its beginning in the practice of a kind of beverage with snow from snow-capped mountains. With the discovery of this phenomenon, Salt Peter made freezing mixtures with ice, which not only chilled but actually froze beverages, resulting in a product like ice cream. The percentage of milk utilization for ice cream is 0.7% of the total milk produced in the country. The growth rate for the ice cream industry was estimated to be 25%- 35% [10]. The richness in nutritive constituents of ice cream has been realized by all, but the problem lies in the production and handling of this food, which is very complex and is associated with many problems. Since ice cream is a milk-based product, it is rich in all milk nutrients and acts as a growth medium for various microorganisms. The growth and multiplication of microorganisms are possible during the production, storage, handling, transportation, and marketing of these products. The contaminating microorganisms in the food are traced in ingredients added and environmental factors such as air, faults in the storage tank, and cracks in the plant and packaging materials [11].

Out of 20 Ice cream samples processed for the isolation and identification of microorganisms. Some samples showed the presence of bacteria. As ice cream is a rich source of milk nutrients, i.e., Casein (Milk protein), Lactose (Milk sugar), added sugars, and cream rich in lipids, it acts as a good medium for growth and multiplication of microorganisms, mainly bacteria and fungi. Good ice creams are nutritious and are not responsible for any ill effects or diseases in consumers. Whereas the contaminated ice creams are mostly responsible for various diseases like sore throat, tonsillitis, typhoid, and food-borne illness [12]. The nutritional value of ice cream is also reduced due to the growth of proteolytic and lipolytic organisms, which utilize the protein, fat, and other nutrients present in ice cream. The sources of contamination of ice cream are many, but the main sources are water and milk. Water used for the preparation of ice cream may act as a main source. The water may contain faecal coli forms and thus may contaminate ice creams. The milk used for the production of ice cream may not be properly pasteurized, which can also be a reason for the persistence of contamination. The other major sources of contamination are utensils and equipment used for the ice cream preparation, air in the plant, and food handlers either suffering from diseases or being in a state of healthy carriers harboring the organisms in the nose or throat. The risk for rheumatic fever, a serious immunological and physiological problem that mostly affects the heart of children, is because of *Streptococcus pyogenes*. One of the main reasons for this disease is the contaminated ice creams. Tonsillitis leads to tonsillectomy (Surgical removal of tonsils. This is commonly seen in children consuming ice cream frequently [12].

## Conclusion

According to the results obtained in this study, there were wide variations in the chemical composition of the analyzed ice cream samples. The values of total dissolved solids/salts showed considerable differences across the samples. In contrast, the pH values indicated that most of the samples fell within the acidic range, which may pose potential risks to human health. The detection of carboxymethyl cellulose (CMC) in the samples is particularly concerning, as most recorded concentrations exceeded 100 ppm, the limit recommended by the World Health Organization (WHO). Microbiological analysis revealed the presence of several bacterial and fungal species in the tested samples. These microorganisms are known to be associated with various diseases and may negatively impact consumer health. Given that ice cream is one of the most widely consumed foods, especially among children, strict storage and transportation conditions are essential to maintain safety and quality. Future research should focus more extensively on both the chemical and biological aspects of ice cream. In particular, studies on the total bacterial count, as well as the isolation and identification of bacterial and fungal species, are highly recommended to better understand the risks and ensure consumer protection.

## Acknowledgment

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## Conflict of interest. Nil

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