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DEVELOPMENT OF PROTOTYPE E NOSE FOR DETECTION OF ADULTERANTS IN RAW MILK

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Abstract: This paper deals with the development of the prototype model of "Electronics nose" hereafter E-nose, for the detection of adulterant in raw and pasteurized milk. As it is well known that nutrients food improves health, whereas adulterated food deteriorates health, people have become more concerned about the food adulteration and spoilage of food. In the present situation, most of the foods are adulterated for economic benefit. This causes health issue results in severe diseases such as indigestion, acidity, ulcers, cancers, malfunctions of kidney and liver, etc. To overcome these problems, novel methods for the detection of adulteration of food and milk have become important. The role of the Internet of Thing (IoT) in this context is also included in this paper for easy detection of milk adulteration. The method is explained along with the experimental investigation using certain samples of raw and pasteurized milk. The result indicates the effectiveness of the method in classifying the adulterated milk samples. The detection of adulteration estimated by this work has been reported better than the other previous reports.

Keywords: Electronic nose (E-nose), Graphical user interface (GUI), Internet of thing(IoT), Sensor array, Linear discriminant analysis (LDA), Internet protocol(IP)

I. INTRODUCTION

In the current scenario, food safety is one of the emerging and important global issues with international trade and public health implications [1]. In developing countries, the concerns about food safety are increasing due to a variety of factors including the increase in the age of human populations, unplanned urbanization and migration and mass production of food due to population growth and changed food habits [2]. Milk is one of the foods that have serious challenges in its handling and distribution [3]. Milk is one of the nutritional food consume worldwide due to its widely known nutritional value and health benefits according to Fox et al [4]. Apart from being a source of nutrients essential for growth, development and maintenance of health, it is a major source of protein in the diet of young animals and humans of all ages. According to adults can also benefit from drinking milk, to help keep their bones strong which will reduce the risk of developing osteoporosis.

Adulteration of milk is one of the social problem exists in both developed and developing countries. This is due to lack of regulations or enforcement, proper refrigeration techniques, high yields with no market and hence the use of high levels of different adulterants toelongate the shelf life, prevent spoilage, increase thickness and whiteness.

In India, commonly added adulterant is urea for milk, which decreases the nutrients of the milk. Urea is chosen to adulterate the milk since it is cheap and rich in nitrogen [6]. More urea present in the milk may cause indigestion, acidity, ulcers, cancers, malfunctions of kidney, etc. After addition of water to the milk will reduce the density of the milk.

In order to increase the density of milk, sometimes Chlorine is added to the milk. Mastitis in cow also raises the chlorine level in the milk. It will cause heart problem and clogging in arteries. Like urea, other adulterants such as, Formalin, Hydrogen peroxide, Liquid whey, Water are also commonly used adulterants and their effect is also as harmful as urea. Hence adulterants estimation in milk is of great significance [3].

The traditional techniques have drawback like time consuming, skilled workers and expensive equipment. The Diary industries require non-invasive, fast, reliable and low-cost device to detect the milk adulteration for human health concern. The E-nose is a rapid and non-invasive method of identifying the aromas and it may be used to detect the quality for food product. The E-nose is a novel method to control the quality of dairy product and it is not being used. Hence an attempt has been made to design and develop a prototype E- nose for detection of adulterants in milk [2].

II. LITERATURE SURVEY

Adulteration of milk detection is the determination of the presence of adulterants in milk. Analysis of milk aroma is the best promising tool to obtain information about the milk and one of the best methods to isolate the flavor compound in headspace analysis. Developed an ultrasonic embedded system for detecting water adulterated with milk by varying fat contents and varying the different percentage of water added to milk [8].The developed system using near-infrared diffuse reflection spectrometry used to detect the water adulterated with milk and show 99% accuracy[9].

Kandpal *et al* [10], a specific gravity of the milk was assessed by lactometer and chemically tested by adulteration test kit. It was observed that out of 60 samples of milk, only 12 samples (20%) had a specific gravity of 26 and more which is considered as undiluted milk while 48(80%) had a specific gravity of less than 26 clearly indicating the dilution of milk with water. Aziz amari *et al*[11] employed simple electronic nose devices to discriminate among raw milk three different dairy farms. And also, to perform a characterization of one raw milk in different aging days. The employed sensor array consists of six different Taguchi gas sensors (TGS) and support vector machines (SVMs).

Volker Muller et al [12] studied the process of bacterial fermentation under anaerobic conditions, in the dark and in the absence of electron acceptors. Various types of fermentation and determined that ethanol is the major product of the anaerobic fermentation are explained. This paper also stated that lactic acid is the common end product of fermentation and determined various bacteria that are responsible for the variation of different chemicals in milk. Among bacteria, fermentation is found in a number of organisms belonging to very different phylogenetic tribes. It is not restricted to morphological groups, a pH range or salt concentration.

Santosh M. Tambe *et al* [13], are common adulterants found in milk are urea, starch/blotting paper, glucose/sugar, caustic soda, ammonia refined vegetable oil (cheap cooking oil), white paint and common detergent or shampoo. It is having content such as glucose, PH in the milk sample and the sodium chloride is detected.

Renny *et al* [14] proposed an enzyme-based sensor for detection of urea in milk was constructed using a piezoelectric sensor, which measures the pressure of the gas, evolved in the sample. The sensor showed linear behavior for varying concentrations of urea in the samples. The results indicate that this technique can be effectively used to detect urea levels in milk. Hemanth Singuluri et al [15] studied was carried out keeping in view the recently emerging concern of adulteration of natural milk with various illegal substances to increase its marketability. This study explains in detail the hygienic status of milk supplied to various cafes, small hotels, and other public and educational institutions.

Collected milk and milk products samples collected were adulterated with common adulterants like water, urea, detergent and starch, water being the most common adulterant [16]. Hui chun Yu *et al*. [17] monitored the adulteration of milk with water using an Electronic nose containing 10 different metal oxide sensors. Whole fluid milk adulterated with different proportion of water is examined and also compared the results obtained by principal component analysis and linear discriminate analysis.

III. MATERIALS AND METHODS

More than 100 samples of fresh HF cow raw milk and pasteurized milk were collected from different vendors and also local Government outlets (Dairy) at Mandya (Karnataka) and nearby villages. About 100ml raw and pasteurized milk samples have been considered for the experiment. The raw milk samples later mixed with adulterants such as hydrogen peroxide, formalin, and urea.

Conventional methods such as chemical testing, gas chromatography, and commercial Electronics nose were used for the detection of adulterants in milk. The drawback in earlier methods was time-consuming, untrained persons for the use of equipment, etc. In order to overcome the above drawback, a novel method was proposed in this research work and this paper is about the basic setup for E-nose for investigation of adulteration of raw milk.

IV. EXPERIMENTATION

The proposed embedded system consists of hardware and software which can detect the adulteration of raw and pasteurized milk.

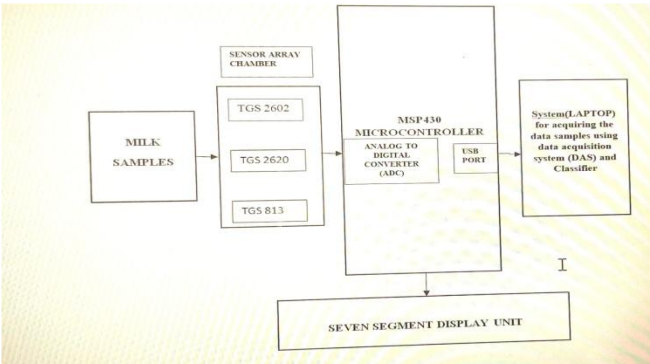

Fig.1 (a): Block diagram of proposed prototype Electronics nose for adulteration detection in milk

The block diagram of Fig.1 (a) consists of a sensor array chamber, MSP430 microcontroller, and data acquisition system together with a display for visual indication of the adulteration in the sample of milk considered. The proposed system is designed such that it will display the to classify the quality percentage of adulteration in the milk. Further, the system is helpful of milk.

Fig.1(a) shows a block diagram of the proposed prototype model of E-nose. It consists of various subsystems as explained below.

Collection of samples: Every ten samples of 100ml of raw milk of Holstein Friesian (HF) cow and raw milk added with different volume of adulterants like hydrogen peroxide(H_2O_2), formalin and urea are prepared in a 500 ml peroxide(H_2O_2), formalin and urea are also prepared in a beaker. Similarly, the 10 samples of 100 ml pasteurized milk and milk added with different volume of adulterants like hydrogen 500 ml beaker.

Sensor array chamber: The sensors used in this system are MOS sensors such as TGS 813, TGS2620 and TGS 2602. These sensors are used to detect formalin, H_2O_2, and urea. The sensor array is formed by connecting each one a detector circuit and then it is placed in the sensor chamber which is an airtight box. This chamber consists of a sensor array, temperature and humidity sensor.

The sensor arrays are placed on top of the sensor chamber, where the samples are placed inside the chamber. It is placed in such a way, the sensor array is 3m above the surface of the sample in a chamber. The sensor array absorbs the volatile organic compound (VOC) of the samples and this makes the resistance changes in the sensor which equivalent to the gases react with the sensor surface. These resistance changes in the sensor are detected by the detector circuit which gives output in term of voltage.

MSB 430 and data acquisition system: MSP 430 microcontroller are interfaced with the detector circuits of the sensor array to acquire the data samples, then process and record the data using a data acquisition system. The sensor array output for the raw milk and pasteurized milk are recorded for each sample. Based on the reading of the samples, the threshold value of each sensor is found. The program is written for three sensors for setting the threshold value in the microcontroller.

In real time, milk samples are placed in a sensor arrays chamber and sensor array takes 3.5 minutes to get stable responses. These outputs from the detector circuits acquired and then processed by the microcontroller. If anyone sensor output goes above the threshold value of the raw milk. Then microcontroller will display on the monitor as "**Milk is Adulterated",** otherwise as "**Milk is not Adulterated**".

The data acquisition system is done in the personal computer system, graphical user interface (GUI) is created in the system so that three sensor readings, as well as testing of the milk adulteration, can be done in real time from anywhere in the world using the Internet of thing (IoT). Using the IP address of the network, we can access and test milk samples anywhere in the world either using a mobile or system by pressing the test button in the GUI and it will display the sensors outputs, temperature, humidity and also milk is adulterated or not.

The fig.1(b) shows the sensor model setup for the experimentation and the interfacing of the MSP430 microprocessor to use it as an IOT. The pH meter and a temperature sensor for the detection of adulterants are as connected in the above fig.1 (b). The threshold of pH value for the normal milk is 6.5 and it is compared with the pH value obtained from the sample of milk tested.

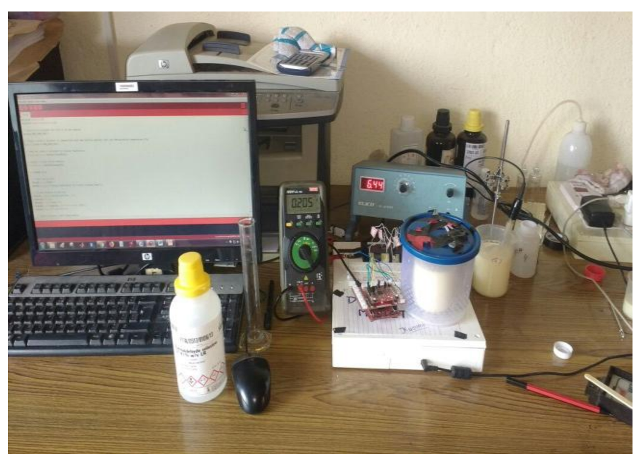

Fig.1(b): **Experimentation model of Prototype E-nose**

V. RESULT AND DISCUSSION

The following are key observations from the experiments conducted:

In 100ml raw milk, urea concentration is between 15.8 to 20.56 mg/dl is normal and the above these are adulterated milk [6]. The 0.6ml of formalin can be added to the 100ml raw milk as preservatives safe for human health, in order to increase the shelf life of milk. Excess of formalin added to the raw milk causes health issues such as kidney and liver damage [7].

Discrimination of raw and pasteurized milk:

100 ml of raw milk samples of HF cow in a beaker placed in a sensor array chamber, sensor responses are recorded. These outputs of the sensor array are recorded and plotted as shown in fig.2. The fig.2 gives the pattern of the raw milk of HF cow and also gives the compositions like hydrogen peroxide is 0.039V, formalin is 0.068V(0.6 ml) and urea in term of ammonia is 0.152V(15.8 mg/dl in 100 ml milk). The same samples are applied many times to the sensor array chamber, the output of each sensor gives the same responses.

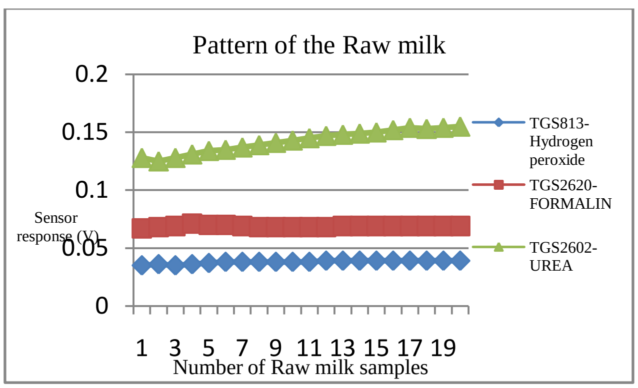

Fig. 2: **sensor response for natural milk**

From these, we can conclude that the threshold value of TGS 813 (H_2O_2) is 0.039V, TGS 2620 (formalin) is 0.068V and TGS 2602 (Ammonia) is 0.152V are found in raw milk. If any of the sensor responses goes above the threshold value, milk is considered as adulterated.

Take 100 ml pasteurized milk samples in a 500 ml beaker and placed in a sensor array chamber. TGS 813, TGS 2620 and TGS 2602 give responses as 0,039V, 0.068V and 0.228V. These responses are plotted as shown in fig.3 and give the pattern of the pasteurized milk and also gives the compositions like hydrogen peroxide is 0.039V, formalin is 0.068V(0.6ml) and urea in term of ammonia is 0.228V. The same samples are applied many times to the sensor array chamber, the output of each sensor gives the same responses.

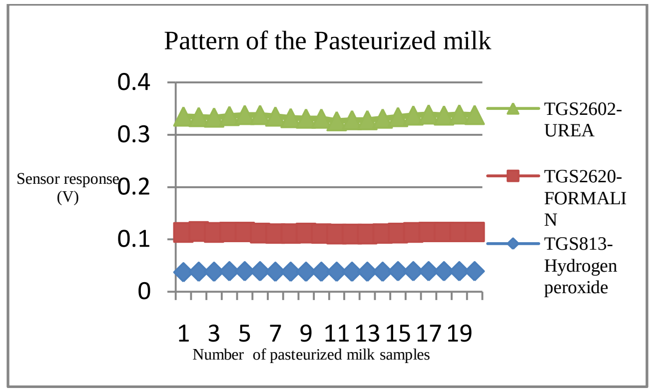

Fig.3: **sensor response for pasteurized milk**

TGS 813 used for the detection of Hydrogen peroxide in milk. Since hydrogen peroxide is used as a preservative in areas where refrigeration is not available, the sensor array subjected to raw milk and pasteurized milk samples results nearly like baseline readings. As raw milk and pasteurized milk does not contain hydrogen peroxide. TGS 2620 is used for the detection of formalin and the formalin increase the shelf life of milk. Naturally, there will be a certain amount of formalin present in milk as it extends the quality of life of milk. TGS 2602 detects urea in milk and urea provides whiteness and increase consistency of milk.

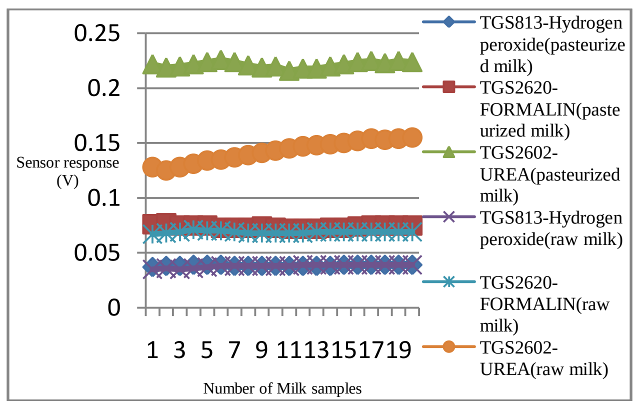

Fig.4: **Comparison of sensor response for raw and pasteurized milk**

Fig.4 shown the plotting of raw and pasteurized milk sensors responses, from this fig.4, we can conclude that pasteurized milk contains more urea compared to raw milk since the amount of urea varies with different breeds of milk in pasteurized milk. Since there is no presence of hydrogen peroxide in both the milk samples, they are nearly appeared to baseline readings. The amount of formalin in pasteurized milk is slightly higher than the raw milk since the pasteurized milk is preheated; shelf life is more compared to raw milk.

For the detection of adulterated milk, the threshold value fixed by comparing it with raw milk sensor response and to verify the volume of chemicals inside the milk, a specified amount of chemicals added to the milk and sensor response are noted. Once the analysis did the threshold for each volume of chemical is specified for all chemicals according to sensor response.

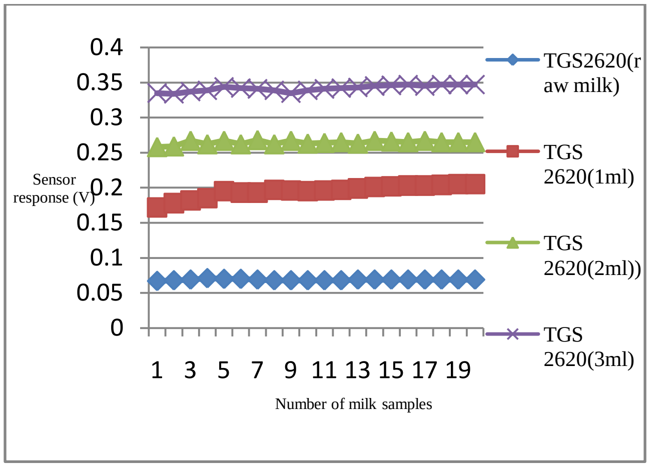

Fig.5: **Sensor response to the different volume of formalin added to raw milk**

Fig.5 shows the sensor response for the different volume of formalin added to the raw milk sample for the analysis of sensor reaction to different concentrations of chemicals added to raw milk. From this figure.5, we can conclude that the threshold value of the different volume of formalin added to the raw milk is set and also clearly distinguish between the amount of formalin in raw milk and the raw milk adulterated by different volume of formalin (1ml,2ml, 3ml) can be observed. As the concentration of formalin increases sensor response is also increases. Since the formalin added to milk to increase the shelf life of the milk, when it crosses a certain level it affects the kidney and liver of the human body.

Fig.6 shows the sensor response for the different volume of hydrogen peroxide added to the raw milk sample. Since the hydrogen peroxide used where refrigeration is not used in rural areas in order saves the cost of electricity. From these fig.6, we can conclude that the threshold value of the different volume of H_2O_2 added to the raw milk is set and also clearly distinguish between the amount of H_2O_2 in raw milk and the raw milk adulterated by different volume of H_2O_2 (1ml,2ml, 3ml) can be observed. As the temperature of the milk decreases and the PH of the milk increases when the different volumes of H_2O_2 added to the raw milk samples.

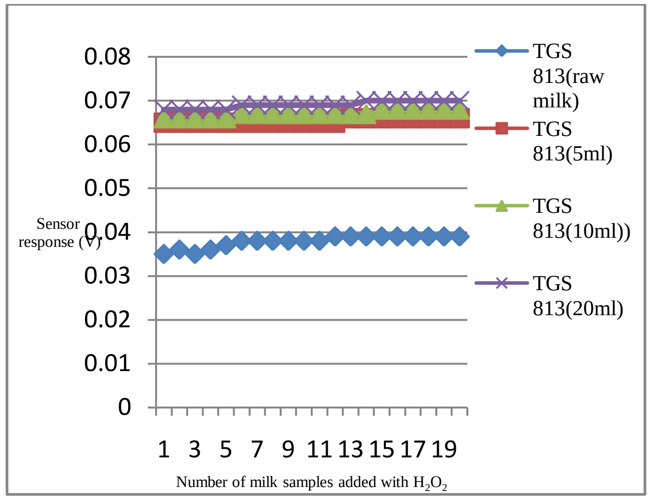

Fig.6: **Sensor response to the different volume of hydrogen peroxide added to raw milk**

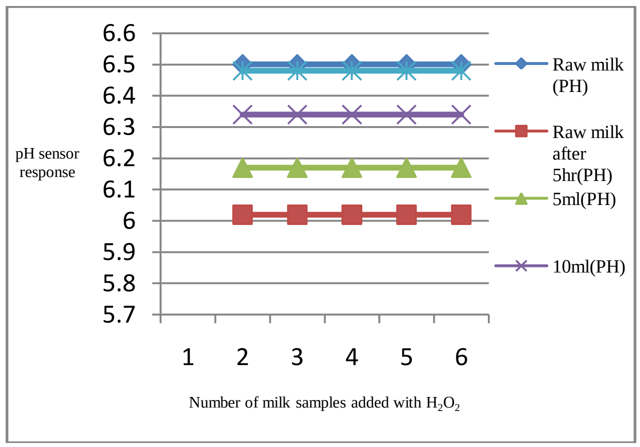

Fig.7: **pH Sensor response to the different volume of hydrogen peroxide added to raw milk**

Fig.7, shows clearly distinguishes the pH level for a different volume of H_2O_2 added to the raw milk, when hydrogen peroxide added to it, the pH level increased hence the shelf life of the milk increased. Intake of hydrogen peroxide when it reaches above threshold value causes reduces fluid intake, food consumption and reduced the body weight of the human body.

From the experimental results show in the fig.8, it can be concluded that the threshold value of voltages from the sensor for a different volume of urea added to raw milk will vary linearly.

The same values have been set in the microcontroller based setup to verify the milk adulterated by urea. This has been verified by conducting the experiment as above and the result is found to be satisfactory. Further, it also is noted that the higher value of urea added would damage the human organ and hence this experiment gives the basic result to develop the E-nose to detect the milk adulteration.

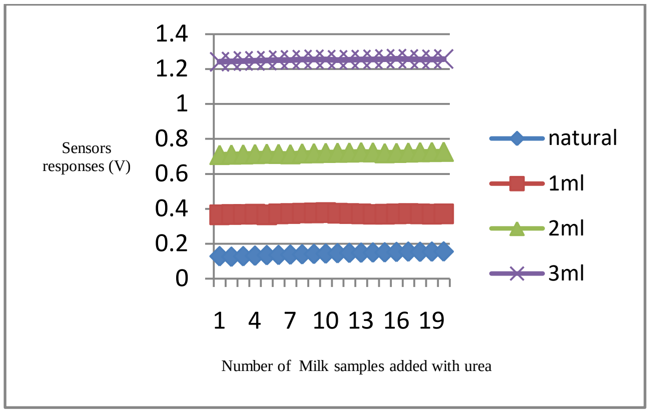

Fig.8: **Sensor response to the different volume of urea added to raw milk**

According to the threshold values set for urea, formalin and H_2O_2 added to raw milk for the identification of adulterated milk samples and the program is written in MSP430 microprocessor in order to use it as IoT.

Fig.9 shows the GUI in the system to test milk is adulterated or not by using internet protocol(IP) address from anywhere in the world. When the sensor responses from sensor array chamber are interfaced to the microcontroller through analog to digital converter (ADC) and the reading are compared with the threshold value set, if it exceeds the threshold value set, it will display message on the GUI, Milk is adulterated, otherwise it will show Milk is adulterated with 1ml of urea as shown in figure.10.

Fig.9: GUI in the system for Milk Adulteration Detection testing page

Fig.10: Detection of 1ml urea present in milk as Adulterants

In order to the validation of output responses of a sensor array, we are using linear discriminant analysis (LDA) for the classification of raw milk and adulterated milk. The result obtained is as shown in fig.11. Later the results obtained from sensor array are applied to the LDA classifier and the classification of sensor response is as shown in fig.11.

Similar experimentation with pasteurized milk as well as different breed milk added with different volume of adulterants such as urea, formalin, H_2O_2 was conducted and our proposed system is able to clearly distinguish between milk adulterated and milk not adulterated.

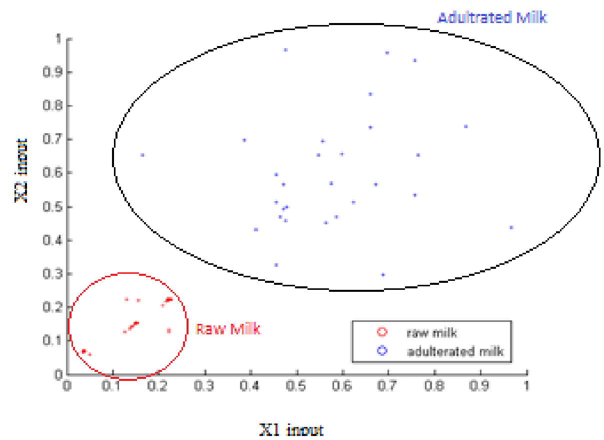

Fig.11: **Classification of raw milk and adulterated raw milk samples using LDA**

VI. CONCLUSION

In this paper, an attempt has been made to develop a prototype E-nose for the detection of adulterants in raw and pasteurized milk. The methodology adopted has given a better response for milk adulterants by the use of MOS sensors. The results have been validated using LDA with approximations of 90 % accuracy. Further, this technique may be employed for the detection of bread, Existing standard for adulterants detection in milk is often a rudimentary method, and they normally deal with the detection of adulterants using a chemical method. The method of adulterants detection for milk using gas sensor array shows the variation in concentration of VOCs with respect to time, a difference between raw and adulterated milk in terms of VOC concentration. This classification gives a clear picture of the milk that is more adulterated. Adulterants detection using a gas sensor array has minimum procedural steps, easy to handle, gas sensors are economical. This method can be implemented in the dairy industries to avoid mixing of adulterated milk with other milk. Schools and Montessori's can implement this method to detect milk adulteration.

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