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Milk fraud by the addition of water using an ultrasonic echo-pulse technique

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Abstract

Milk is one of the essential food commodities and plays a crucial role in the diet of human nutrition. The quality of milk has been challenge in industry due to the threat of adulteration. Adulteration in food products has many aspects such as the addition of prohibited substance either partly or wholly for the state of financial gain, the lack of hygienic conditions of processing and storing, the decreasing of the amounts of original ingredients, or the changing of their chemistry itself which leads to the consumer being cheated. Ignorance of this fact is not fair since this may endanger consumer health. With the aim of monitoring milk quality at distribution points, a non-destructive pulse-echo method was proposed to evaluate the water content in powdered milk. The results indicate the ability to measure the adulterant level based on the value of the peak to peak amplitude at room temperature. In addition, the technique that is developed in the present work involves only one transducer which is non-invasive and considered as a very important in the food industry for hygiene reasons.

Keywords: Adulteration, peak to peak amplitude, powdered milk, ultrasound, water content

1. INTRODUCTION

The milk considered as a complete diet because it contains the major elements required for growth and production like Lactose, Fat, Protein, mineral, and vitamins in balanced ratio rather than the other foods as mentioned by Fox (1992).

In the dairy industry, milk quality is measured by criteria such as lactose, fat, protein, and water content. This later influences storing conditions and shelf life as well as textural and technological qualities. On the other hand, when pure milk is adulterated, its physical properties change, such as electrical conductivity, electrical admittance, boiling point, freezing point, viscosity, etc.

In the last years, contact-based methods have been used in order to detect adulteration present in milk. These methods include chemical reactions on the milk to detect adulteration (Cozzolino et al., 2001). Although these methods produce results, the samples cannot be reused, resulting in a waste of milk. Also, the amount of sample required is large, so this method is not always applicable. In addition to chemical methods, many researchers have contributed to different types of destructive techniques (Pavić et al., 2002; Gnan et al. 1982: Cady et al., 1978). E-tongue (Dias et al., 2009; Paixão et al., 2009) is one of the most recognized methods of adulteration detection and it reached a commercial level. It used 36 cross sensibility sensors allowing successful recognition of 5 basic taste standards. Although it detects many types of adulterants, the process is time-consuming and not very accurate. Later non-contact methods like spectroscopy were proposed which can retain the sample for further use (Kasemsumran et al., 2007; Haibo et al., 2008). However such methods suffer from a lack of accuracy and hence are not always reliable.

In order to address these problems, the ultrasonic method is used. In this sense, several works, in our laboratory, have involved this technique for evaluating the quality of food products, such as milk

(Ouacha et al., 2015; Derra et al., 2017; Bakkali et al., 2017), water (Hamine et al., 2010), oil (Izbaim et al., 2010), fish (El Kadi et al., 2013) and orange (Aboudaoud et al., 2012).

Nevertheless, in the field of milk adulteration, the number of papers is still very limited. For example, (Dave et al., 2016) used two parameters, the attenuation coefficient and time of flight to analyze the fat content and the amount of water adulteration present in raw milk samples obtained from different dairies and skimmed milk samples with different fat content from popular Indian brands. Also, (Nazário et al., 2008) applied neural networks techniques to classify milk samples as a function of the amount of water and serum added to milk, relating these properties to acoustic parameters (Ultrasonic propagation velocity and attenuation coefficient) as a function of temperature. Finally, (Elvira and Rodriguez, 2009) showed that acoustical characterization of tainted liquid milk, through density and/or sound speed measurements, can be used to detect gross melamine contamination.

The aim of the present work is to develop a new ultrasonic measurement system providing a non-destructive determination of water content in powdered milk based on peak-to-peak amplitude.

2. MATERIALS AND METHODS

2.1. Materials

Milk samples were prepared by dissolving 13g of whole milk powder from an international commercial brand (contains 26% of fat content i.e 3.4% of fat content after reconstitution and 24% of protein) in 90mL of warm water (These are the standard conditions).

2.2. Methods

2.2.1. Ultrasonic system

An ultrasonic system was designed to set-up the experimental pulse-echo shown in Figure 1. The milk sample is enclosed in a parallelepiped vessel with 1cm spaced sidewalls. The container input and output side plates are made from plexiglas and glass materials. An ultrasonic transducer of frequency 5MHz and a 10mm crystal diameter (A309S-SU

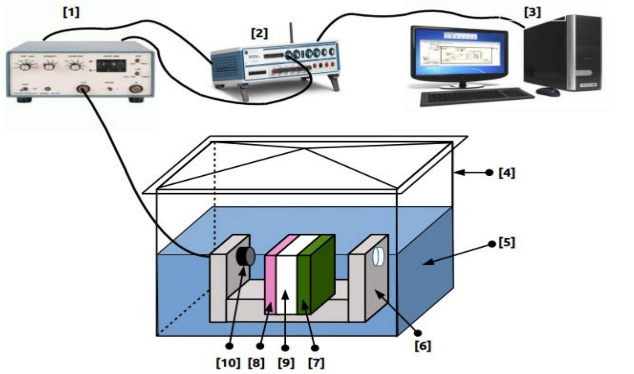

Figure 1. Ultrasonic experimental characterization system

Model Panametrics Olympus) is placed next to the plexiglas side. The transducer played the role of an emitter/receiver; it is connected to a pulse generator (Model 5052P R Sofranel, Voltage 230V, Bandwidth 50MHz). This later excites the transducer via an electrical short pulse which is transformed into an acoustic wave. The received signal (Figure 2) is amplified and digitized by a VirtualBench (Model VB-8012). The measurements were analyzed using a LabVIEW program implemented on a personal computer.

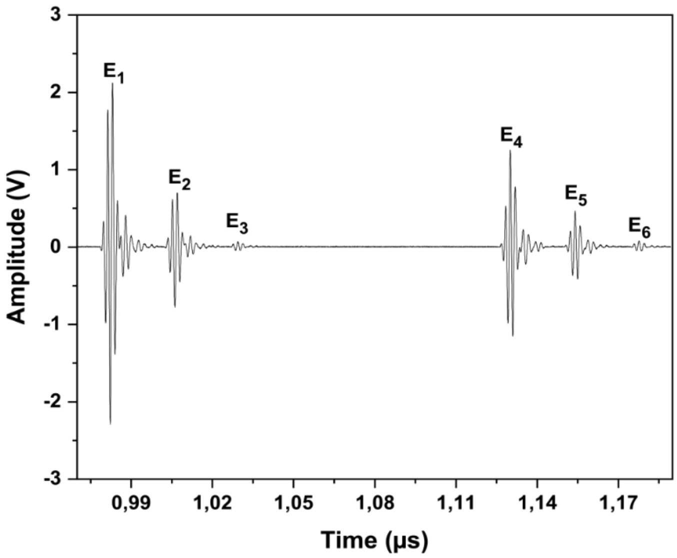

Figure 2. Time domain backscattered signals from milk sample.

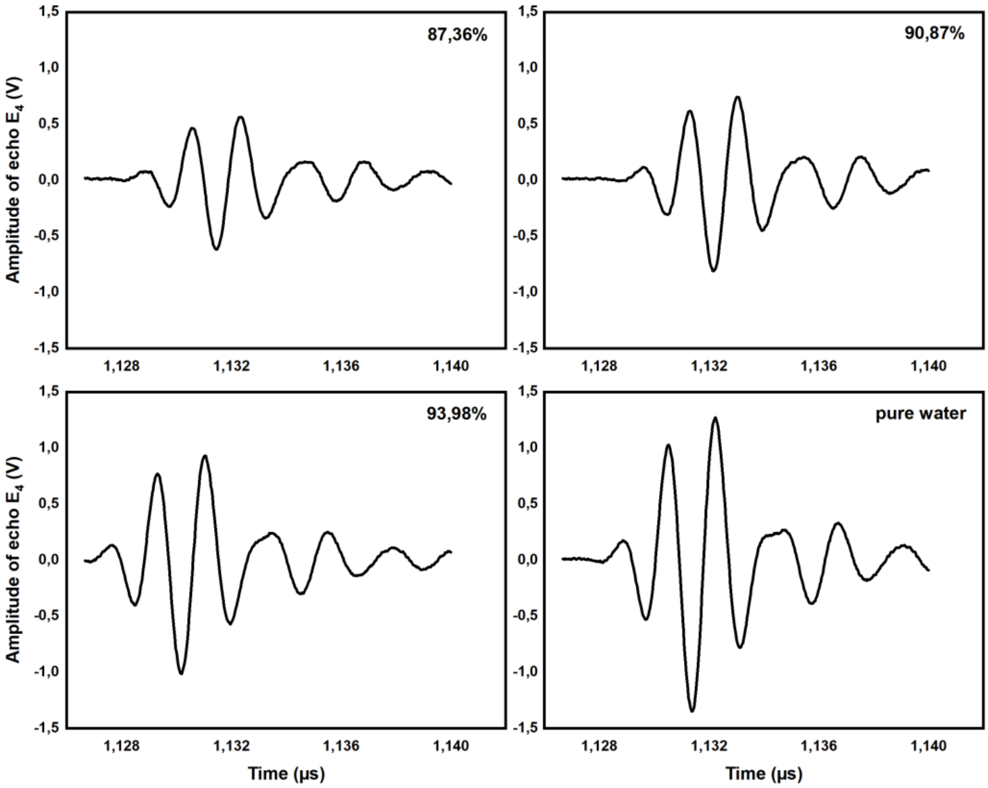

Figure 3. Echo E4 for unadulterated milk sample (87.36%), adulterated milk sample (90.87% & 93.98%) and pure water.

To neutralize noised signals, 25 pulses were averaged for each measurement. The results are composed of two parts: the first part (E1 to E3) is the multi-reflection at plexiglas vessel side in front of the transducer; the second part includes echoes (E4 to E6) having traversed the enclosed milk as indicated in Figure 2.

2.2.2. Experimental design

Samples with different water contents were obtained by adding various amounts of water to the powdered milk. Following the rule of three, the water content in the milk powder, under standard conditions, is 87.37%. During the experiments, milk adulteration was performed by blending variable amounts, ranging from 87.36% to 93.98% (by a dose of 5mL) in a volume of water into 91mL of the original milk sample.

2.2.3. Measurements technique

The reflection method uses a pulse that is reflected at the boundary of known material and the material to be investigated. If a plane wave strikes at normal incidence the interface between two media (M1, M2) with different acoustic impedances, part of the incident wave is reflected back and part is transmitted into the second medium. The peak-to-peak amplitude, App, of a pulse-echo named E, is given by:

$$App = Max\,[A\,(E)] - Min\,[A\,(E)]$$

with A(E) is the amplitude of the pulse-echo whose maximum and minimum are denoted Max, Min.

3. RESULTS AND DISCUSSION

A non-destructive and non-invasive pulse-echo was proposed to monitor in real-time the peak-to-peak amplitude of echo E4 in order to determine the water content in powdered milk.

Figure 3 shows echo E4 for unadulterated milk sample (87.36%), adulterated milk samples (90.87% and 93.98%), and pure water. Based on this figure, it can clearly be observed that the peak-to-peak amplitude of echo E4 increases and get closer to the peak-to-peak amplitude of echo E4 of water (App (E4) = 2.64 Volts) as the sample was more diluted. Besides, the peak-to-peak amplitude measurements are clearly sensitive to the effect of adding water. Moreover, the evolution of the echo E4 presented in the Figure 3 shows that the peak-to-peak amplitude is directly related to the attenuation coefficient. In other words, the smaller the peak-to-peak amplitude is, the greater the attenuation coefficient is. Therefore, milk in its standard state is more attenuated than diluted milk. It is then obvious that water is less attenuated compared to milk since this latter contains water and other ingredients.

The experimental results of the sample peak-to-peak amplitude of echo E4 evolution are shown in Figure 4 at room temperature (the temperature was held at room temperature throughout the experiment to avoid experimental problems and to make these results more widely accepted) with different amounts of water. From the result, it is found that the peak-to-peak amplitude of echo E4 increases with the percentage increase in adulteration. The regression equation obtained after the approximation of the measurement results took a shape of a straight line y = 0.11143x – 8.54365. A high value of determination coefficient R2 = 0.99632 confirms the right choice of the adopted mathematical model. This later allows to quickly measure the level of adulterant depending on the value of the peak-to-peak amplitude.

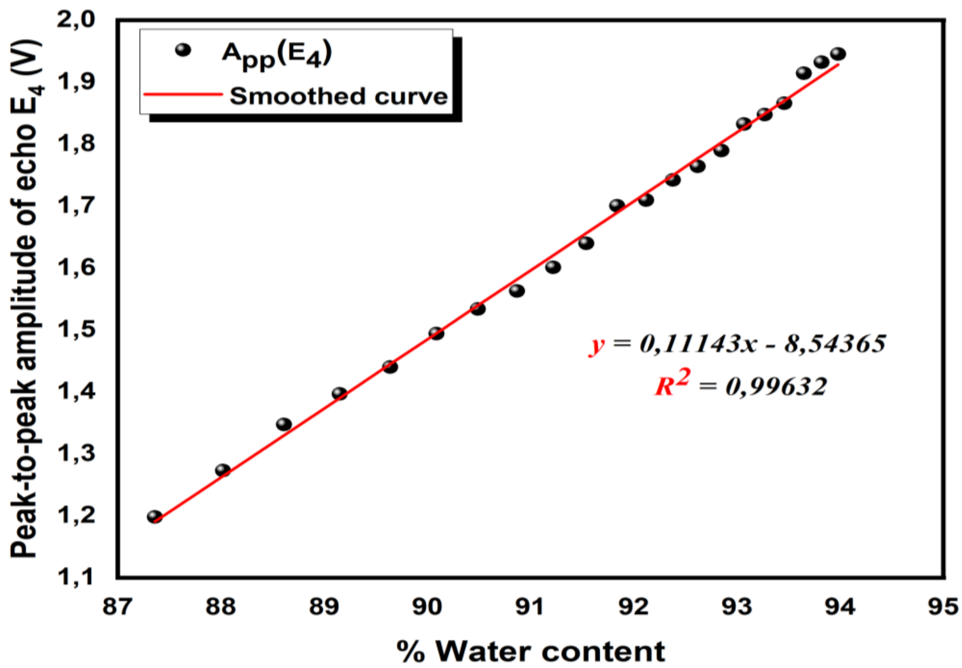

Figure 4. Peak-to-peak amplitude of echo E4 for adulterated milk samples versus different amounts of adulterant

4. CONCLUSIONS

In this study, the ultrasound technique in backscattering pulse-echo mode was demonstrated to be a simple and non-destructive alternative tool to detect milk adulteration with water. This method may prove as a highly efficient and economical method because in this method no chemical is used and the equipment used is also less costly. In addition, This technique can be applied to control the quality of fresh and processed food in packages in the different food processing industries.

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