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A CONTRIBUTION TO THE DEVELOPMENT OF MOISTURE MEASURING OF VACUUM-DRIED HYGROSCOPIC MATERIALS

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Original scientific paper
Dehydration of hygroscopic materials, especially fruit (blueberries, blackberries, raspberries, etc.) and some types of vegetables in vacuum dehydrators by means of radiation at low temperatures preserves their original quality and medicinal value. Experimental research has been carried out, whereby the moisture reduction of vacuum-dried blueberry fruits was measured by applying the capacitive measuring method, in order to check the method and contribute to possible development of applicative devices. A real vacuum chamber with radiating plate electric heaters and additional functional and regulation equipment was used. Measuring sensors were projected and made, and a measuring device was assembled and programmed. By changing very low values of capacitance, the possibility of measuring moisture reduction in blueberry fruits during drying was confirmed. A close connection between the regulation precision of working parameters (subpressure, temperature) in the chamber and the accuracy of measuring the material moisture was underlined. By using an interdigital sensor acceptable sensitivity in measuring the moisture of dried material was achieved. Guidelines for further research and possible development of an applicable measuring device were suggested.

Keywords: *blueberry, capacitive measuring, drying by means of radiation, interdigital sensor, moisture measuring, vacuum chamber*

Doprinos razvoju mjerenja vlage higroskopnih materijala sušenih u vakuumu

Izvorni znanstveni članak
Sušenjem higroskopnih materijala, a naročito voća (borovnica, kupina, malina, itd.) i nekih vrsta povrća u vakuumskim sušarama zračenjem pri niskoj temperaturi, zadržavaju se njihova izvorna kakvoća i ljekovita vrijednost. Provedena su pokusna istraživanja primjenom metode kapacitivnog mjerenja snižavanja vlage plodova borovnice sušenih u vakuumu u cilju provjere metode i doprinosa mogućem razvoju primjenjivih uređaja. Korištena je realna vakuum komora sa zračećim pločastim električnim grijačima i pripadajućom funkcionalnom i regulacijskom opremom. Projektirani i izrađeni su mjerni senzori te je sklopljen i programiran mjerni uređaj. Promjenom vrlo malih vrijednosti električnog kapaciteta potvrđena je mogućnost mjerenja snižavanja vlage plodova borovnice pri provedbi režima sušenja. Istaknuta je uska povezanost preciznosti regulacije radnih parametara (podtlak, temperatura) u komori s točnosti mjerenja vlažnosti materijala. Interdigitalnim senzorom postignuta je prihvatljiva osjetljivost mjerenja vlažnosti sušenog materijala. Predložene su smjernice daljnjeg istraživanja i mogućeg razvoja primjenjivog mjernog uređaja.

Ključne riječi: *borovnica, interdigitalni senzor, kapacitivno mjerenje, mjerenje vlage, sušenje zračenjem, vakuum komora*

1 Introduction

Due to high water content (72 ÷ 95 %), fruit and vegetables are characterized by a low energy value. However, due to the fact that they contain vitamins and minerals, organic acids and fibres, their biological value is high (Fig.1) [1].

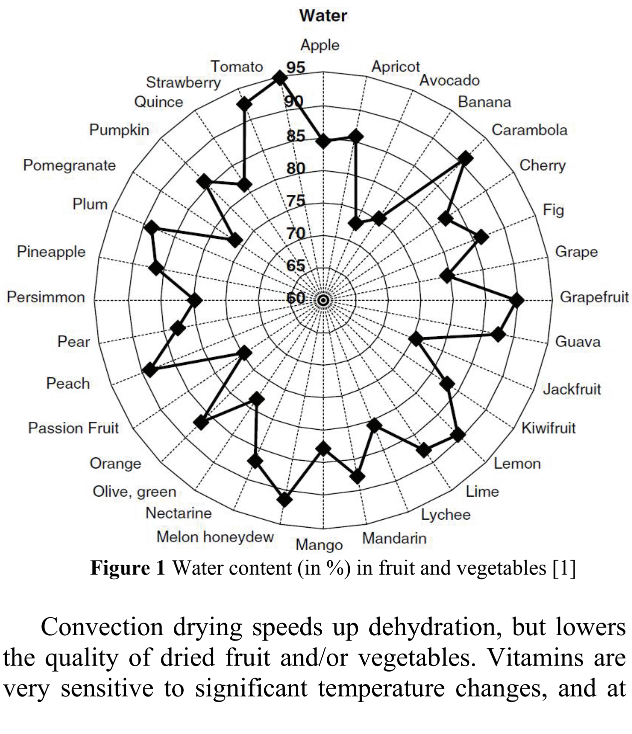


Figure 1 Water content (in %) in fruit and vegetables [1]

Convection drying speeds up dehydration, but lowers the quality of dried fruit and/or vegetables. Vitamins are very sensitive to significant temperature changes, and at higher temperatures they become prone to complete degradation or loss of medicinal features [2]. Therefore, it is necessary to dry medicinal fruit type (blueberry, raspberry, blackberry, strawberry, etc.) by means of radiation in specially shaped dehydrators with high vacuum at low temperatures according to the operating conditions based on lowering the mean moisture content and preserving the quality of dried material.

The mean moisture content of dried material is continuously measured within a closed space without air. According to available data, technological development of sensors used for measuring the moisture of hygroscopic materials (fruit and/or vegetables) in vacuum is quite limited. NIR (Near Infrared), RF and microwave method are often used [3, 4, 5]. The devices are very expensive, unreliable in some measuring conditions or they measure just the surface moisture of a material [6, 7].

Relative dielectric constant of water at the temperature $\vartheta = 20$ °C equals $\varepsilon_w = 80{,}1$ and is 15 or more times higher than the one of a number of other materials (epoxy resin, natural rubber, polyethylene, porcelain, polystyrene, polyamides, etc.). Its value drops as the temperature increases, within range 0 ÷ 60 °C, according to the general relation [8]:

$$\varepsilon_w = 88{,}15 - 41{,}4 \cdot \vartheta + 13{,}1 \cdot \vartheta^2 - 4{,}6 \cdot \vartheta^3, \tag{1}$$

where by the value ϑ - measured temperature value in °C / 100 is inserted. Capacitance C defines the behaviour of two conductive plates at a defined distance when there is electric potential difference between them. Electric capacitance value depends on the surface S and the distance d between the plates, as well as on the type and state of the material that fills in the space between the plates (relative dielectric constant of the material ε_m, $\varepsilon_0 \approx$ 8,854 pF/m dielectric vacuum constant). It is expressed by means of the following equation:

$$C = \varepsilon_0 \cdot \varepsilon_m \cdot \frac{S}{d}, \text{F}. \tag{2}$$

As the material moisture changes, its relative dielectric constant ε_m changes as well, which in constant values S, d and ε_0 affects the value of capacitance C.

Experimental research was carried out, whereby the moisture change of a vacuum-dried hygroscopic material (blueberry) was measured by applying the capacitive measuring method, as a contribution to the development of cheaper applicable sensors and devices with acceptable measuring accuracy within a wide moisture scope of dried material.

2 Object, material and measuring device

2.1 Object and material

A vacuum dry chamber, the useful volume of which amounts to V = 13,6 m^3 ($L \cdot B \cdot H$ = 5 m · 1,6 m · 1,7 m), was used for experimental measuring, Fig. 2. Fresh blueberry fruits with the diameter amounting to 8 mm – 18 mm and with the initial moisture being high $u_p \approx 85 \div 90$ % are placed onto shallow pans made of stainless steel in the form of a layer that is 25 ÷ 35 mm thick. Under a customized regime they are dried until the final moisture content amounts to $u_k = 15 \div 18$ %. By means of sideward plate electric heaters the heat transfer to blueberries is carried out by radiating them under customized vacuum in a chamber $p_v = 850 \div 985$ mbar ($\vartheta = 55 \div 25$ °C).

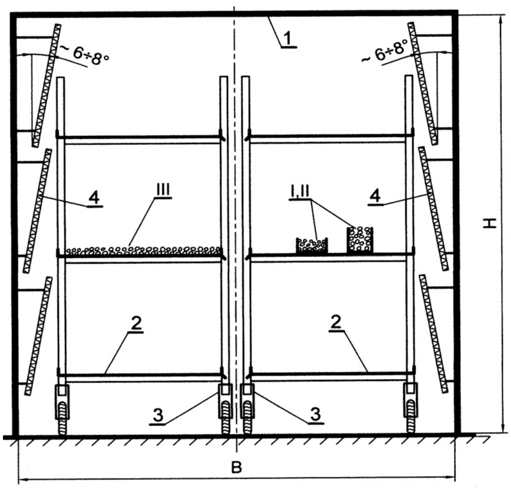


Figure 2 Vacuum chamber – vertical section
(1 – Insolated chamber; 2 – Shallow pans; 3 – Barrow; 4 – Plate electric heaters I, II, III Experiments)

The subpressure in the chamber is achieved by the operation of the main vacuum pump, and the vacuum stability within the anticipated values is maintained by constant operation of the so called "flattening" vacuum pump. After the drying, whole blueberry fruits with skin must not be damaged and nutritive ingredients and vitamins should preserve their original quality and medicinal value. Experimental measuring was conducted in three intervals of drying blueberry fruits from the end of June to the end of July of 2011.

2.2 Measuring device

For the purpose of carrying out experimental measuring of moisture reduction of vacuum-dried blueberry fruits by means of radiation a measuring unit and two moisture sensors were conceived (Fig. 3).

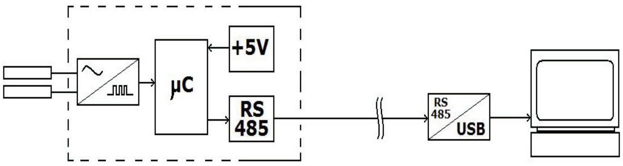


Figure 3 Schematic representation of the measuring device

The measuring unit measures the moisture and the temperature of the material on both channels, the measurement data are transferred and converted through the RS485 communication standard and are saved on the hard disk of a portable computer. While measuring the moisture of hygroscopic material in vacuum it is assumed that the surface S and the distance d between the sensors are constant and that the dielectric constant of the material ε_m changes due to the loss of moisture. As the change in capacitance during the reduction of moisture is small, the electronic assembly has to be very sensitive to the range and accuracy of measured values and insensitive to potential impact factors and disturbances. From the chip line a capacitance-to-digital converter AD7746 was selected [9]. The chip measuring area amounts to ±4,096 pF, i.e. its measuring range amounts to 8,192 pF. Its functional block diagram is shown in Fig. 4.

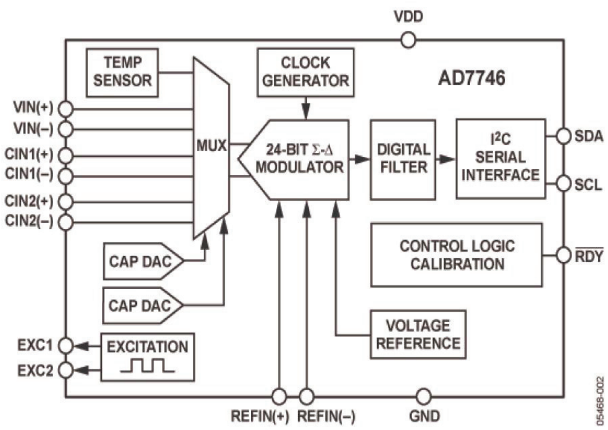


Figure 4 Functional block diagram AD7746 [9]

One and/or two sensors for measuring capacitance are connected to EXC1 and CIN(+) (channel A and/or channel B).

The wiring of the two sensors is shown in Fig. 5.

Based on the conceived wiring diagram and by considering potential impacts (voltage and temperature area, input impulse voltage, operating speed, housing dissipation, noise level, output current, etc.) a two-layer electronic board was projected (Figs. 6a and 6b).

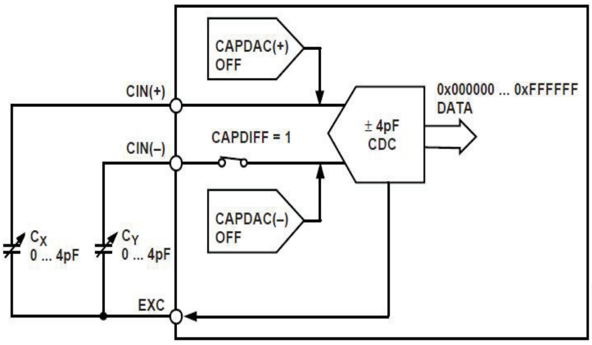
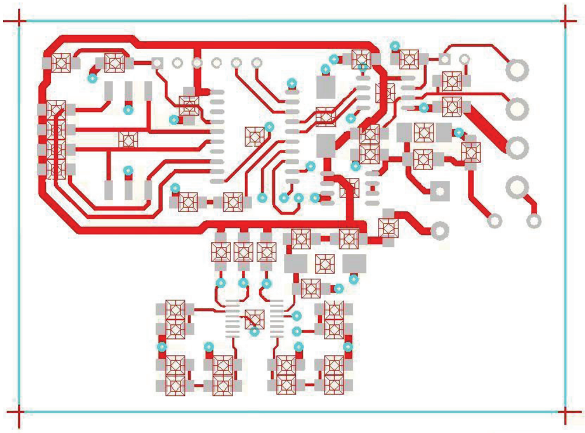
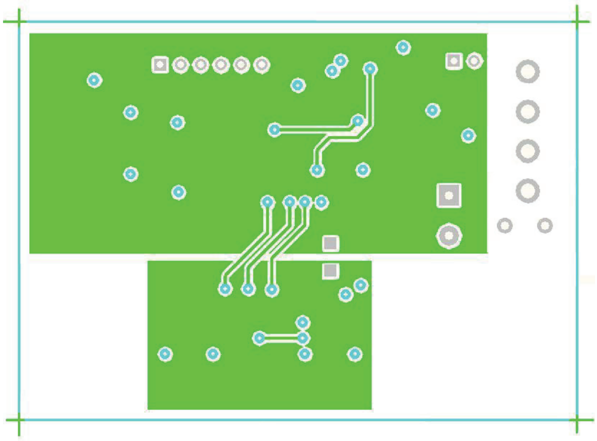


Figure 5 Wiring diagram of the two sensors



a)



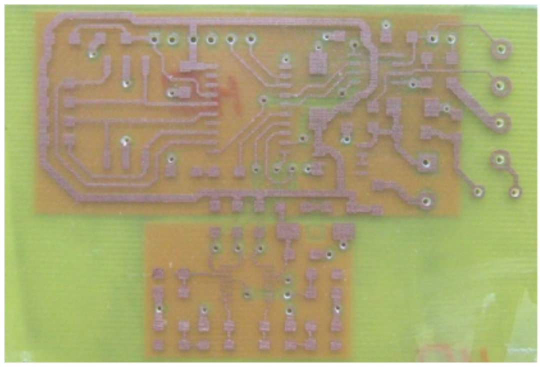
b)

Figure 6 Projected electronic board in PCAD
(a – upper side, b – bottom side)

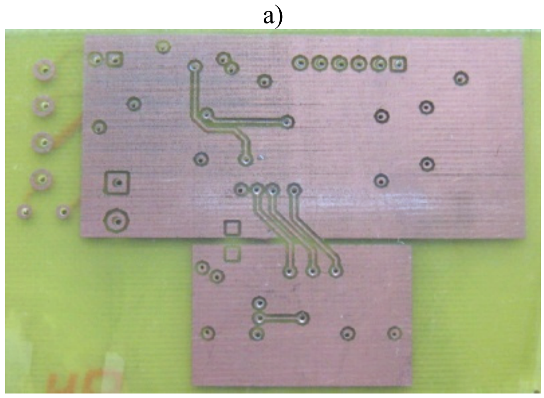
On the upper side of the electronic board components and most of connection lines are situated (Fig. 6a), whereas on the bottom side analogue and digital mass is located (Fig. 6b). The electronic board is made of two-side woven glass fabric with epoxy resin system (FR4) that is 1 mm thick and an 18 μm copper layer by means of a workshop photo-imaging (Figs. 7a, 7b and 7c).

The sensor with opposite electrodes involves two boards (85 × 35 mm) made of woven glass fabric with epoxy resin system (FR4) coated with a thin copper layer on one side. They are glued to the walls of a plastic container that is 95 mm wide (Figs. 8a and 8b).

According to [10] an interdigital sensor was made on a printed glass/epoxy board by using the etching process ($\varepsilon_m \approx 4,7$).



a)



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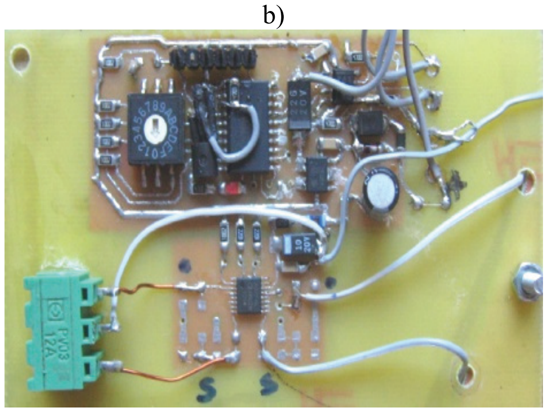
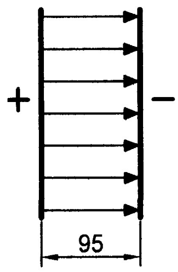
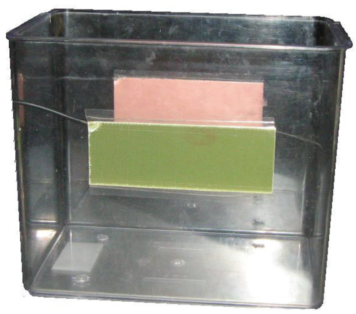


c)

Figure 7 Finished upper (a), bottom (b) and welded (c) electronic board





a) b)

Figure 8 Sensor with opposite electrodes glued to a PVC container

Capacitor plates are positioned in a plane on the printing board. In order to increase the sensitivity and the signal, the capacitor plates (output electrode D and input electrode S) are strips that are 125 mm long and 5 mm wide. By sequential line-up of input and output electrodes with the distance amounting to 25 mm on the upper side of the printing board, the interdigital sensor width was defined. On the bottom side of the printing board, below input electrodes, the protection was carried out for the purpose of neutralizing the electric field below the sensor. The distance between the electrodes defines the penetration depth of the electric field into the material λ that amounts to 60 mm (Fig. 9 and Fig. 10).

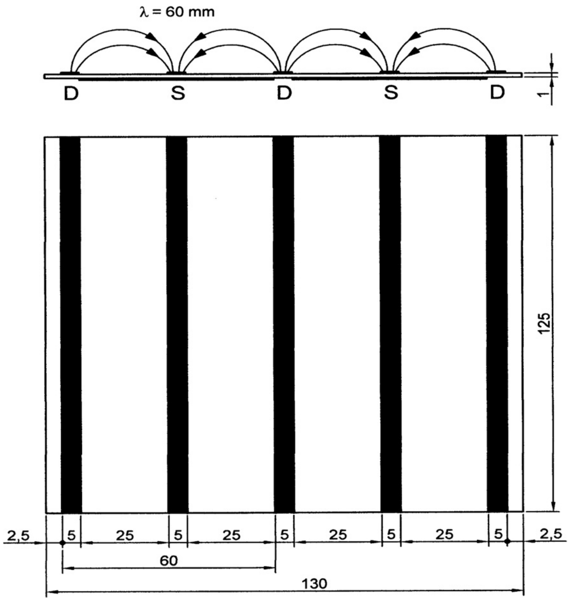


Figure 9 Wiring diagram of the interdigital sensor


Figure 10 Finished interdigital sensor, λ = 60 mm

For controlling the AD7746 chip operation (measures capacitance) the PIC 16F690-I/SO microcontroller was selected [11]. A program in the C programming language was written into it. By using a small amount of blueberry fruits (100 g) at room temperature (24 °C) and relative moisture amounting to approximately 40 %, 20 measuring procedures of reducing blueberry moisture were carried out in intervals of 24 hours with the purpose of checking the operation of the sensor and measuring device. Furthermore, a correction was carried out while writing the microcontroller program. The microcontroller sets the AD7746 chip into a certain operating mode, starts the capacitance conversion and takes over the obtained results. It filtrates and processes these results and classifies them in a form suitable for sending via serial port (RS485) to the portable computer. For statistical processing and graphical representation of the measurement results program packages R [12] and Rstudio [13] were used.

The portable computer was located outside the vacuum chamber and it was connected to the moisture gauge of blueberry fruits within the dehydrator by means of a four-wire insulated cable that was 10 m long.

3 Experiment

The first experimental measuring in the vacuum chamber (I). An electronic board with two sensors was used. The sensor with opposite electrodes (Fig. 11, larger container) was connected to the A channel, whereas the interdigital sensor placed onto the blueberries in the smaller container (Fig. 11) was connected to the B channel. The drive and sense electrodes of the interdigital sensor were directed towards blueberry fruits in the smaller container. The temperature within the vacuum chamber was measured by using the AD7746 chip type. The aim of the first trial measurement was to compare the sensors and determine the functionality of the device in experimental conditions.

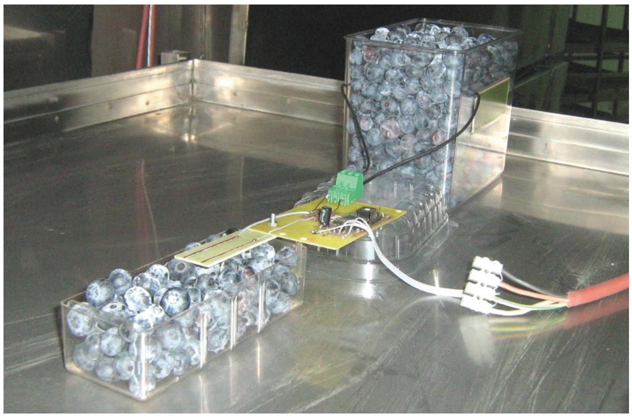


Figure 11 Experimental measuring I and II

Before the drying process started, the mass of the larger container with blueberries had been measured and it amounted to m_1 = 1150 g, whereas the mass of the smaller container with blueberries amounted to m_2 = 350 g. Measurements were registered every 6 seconds and written into the corresponding file. The vacuum chamber drying lasted 385 minutes. Upon the drying completion, by means of digital scales the mass of the larger blueberry container was measured and it amounted to m_{1D} = 1095 g, whereas the mass of the smaller blueberry container amounted to m_{2D} = 332 g.

The second experimental measuring in the vacuum chamber (II). Opposed to the first one, in the second experimental measuring a temperature sensor was added on the electronic board and, according to this, the microcontroller program was adjusted. In this way it was made possible to check the drying temperature value provided by the AD7746 chip. The sensor with opposite electrodes was connected to the A channel, whereas the interdigital sensor was connected to the B channel. The active side of the board with measuring electrodes was placed onto the blueberry fruit layer that was 35 mm thick, whereas the side with the protective electrode was turned towards the area and protected the sensor from the heater radiation impact. Before the drying process the mass of the larger container with blueberries was measured and it amounted to m_1 = 1065 g, whereas the mass of the smaller container with blueberries amounted to m_2 = 320 g. Measurements were registered every 6 seconds and written into the corresponding file. The vacuum chamber drying lasted 440 minutes. Upon the drying completion, by means of the same digital scales the mass of the larger blueberry container was measured and it amounted to m_{1D} = 935 g, whereas the mass of the smaller blueberry container amounted to m_{2D} = 275 g.

The third experimental measuring in the vacuum chamber (III). For the purpose of preserving the fruit quality, blueberry fruits were placed onto shallow pans in a single layer that was up to 35 mm thick and were exposed to the radiation of electric plate heaters and dried in a vacuum chamber up to the final moisture content. During the third experimental measuring the thickness of blueberry fruit layer amounted to approximately 10 ÷ 15 mm. Therefore, instead of the sensor with opposite electrodes, the interdigital sensor was connected to the A channel and it was covered with a single blueberry fruit layer (Fig. 12a).

Figure 12a Setting up the interdigital sensor, A channel

Measurement electrodes were turned towards the blueberry fruits layer, whereas protective electrodes (the other side of the board) were turned towards the bottom of the pan. The same interdigital sensor was connected to the B channel as in the previous measurements and it was placed onto a blueberry fruits layer that was around 10 ÷ 15 mm thick (Fig. 12b). By using scales, the initial mass of fresh blueberry fruits was determined and it amounted to m = 1120 g. Upon drying their mass amounted to m_D = 610 g. Measurements were performed every 6 seconds and entered into a respective file. The dehydration in a vacuum chamber lasted for 400 minutes.

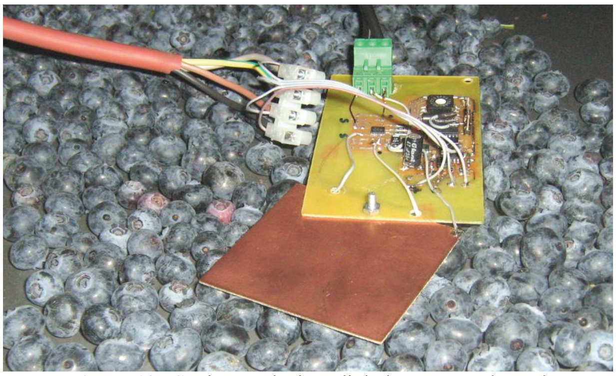


Figure 12b Setting up the interdigital sensor, B channel

3.1 Measurement results

For the purpose of practical representation of measured values results of small changes in capacitance during the number of measurement n, the correlation number k shows the relation 1 k = 0,000488 pF for the measurement area of the AD7745 chip amounting to ±4,096 pF. The value sign of the correlation number k on the ordinate diagram axis (Figs. 13a, 13b, 14a, 14c, 15a and 15c) depends on the sensor and defining the zero position of the measurement beginning within the measurement area.

During the first experimental measuring in the vacuum chamber it was determined that the operation and functionality of the measuring device was correct in all drying intervals. Occasional vacuum oscillation in the chamber between 920 mbar and 975 mbar was recorded. A slight moisture drop of blueberry fruits was noticed. From the blueberry mass in the larger container 55 g of water was isolated, and from the blueberry mass in the smaller container 18 g of water was isolated. The graphical representation of measurement results related to the sensor with opposite electrodes (A channel) is shown in Fig. 13a, and the measurement results related to the interdigital sensor (B channel) are not usable (Fig. 13b). The temperature change curve during the first experimental drying of blueberry fruits is shown in Fig. 13c.

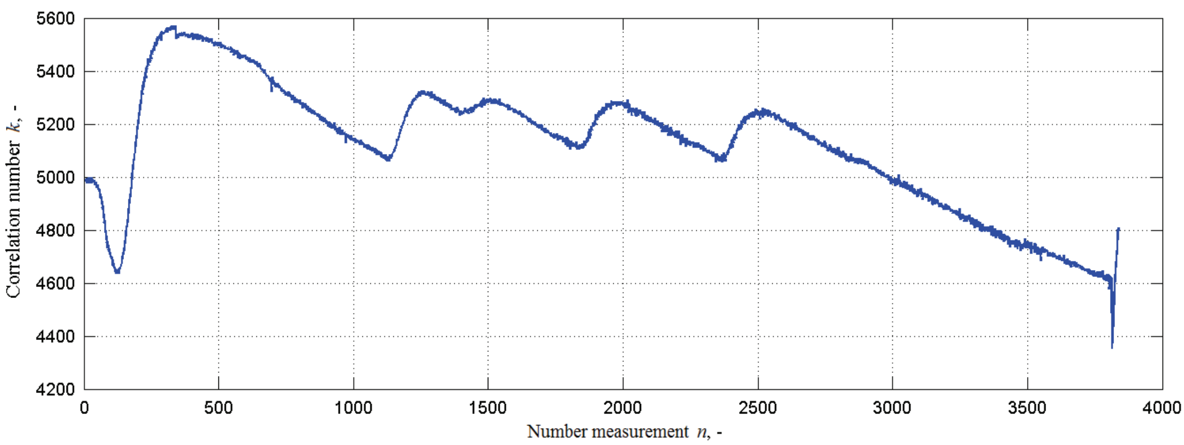


Figure 13a Measurement results – opposite electrodes (A channel), experiment I

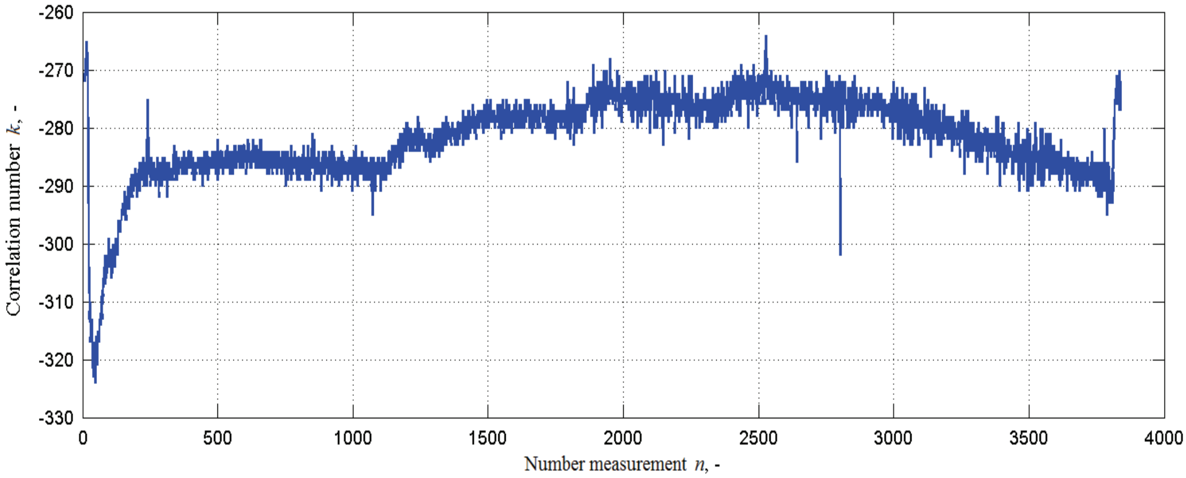


Figure 13b Measurement results – interdigital sensor (B channel), experiment I

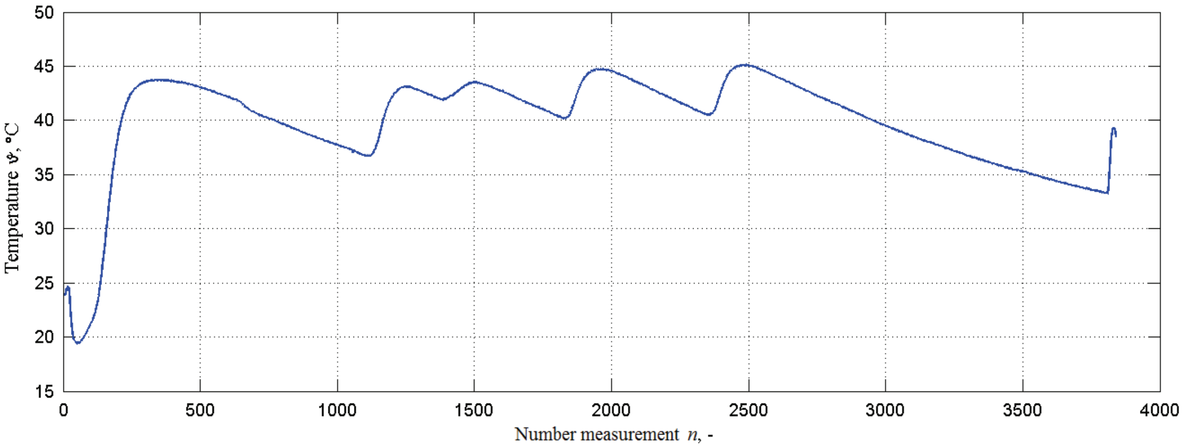


Figure 13c Measurement results – temperature change, experiment I

As vacuum was maintained in the chamber in a more stable manner, the second experimental measuring achieved more usable values by using the sensor with opposite electrodes (A channel), which is shown in diagram in Fig. 14a. A discontinuity of the curve in the interval between 2600 and 3000 number measurement was noticed with the temperature increase (Fig. 14b) resulting from a sudden switch-on of plate heaters. By means of the interdigital sensor (B channel) more precise correlation values of capacitance change at the number of measurement *n* during the drying of blueberry fruits were registered (Fig. 14c), without an expressed sensitivity to the internal temperature change. During the drying 130 g of water was isolated from blueberry fruits in the larger container and 45 g of water was isolated from blueberry fruits in the smaller container.

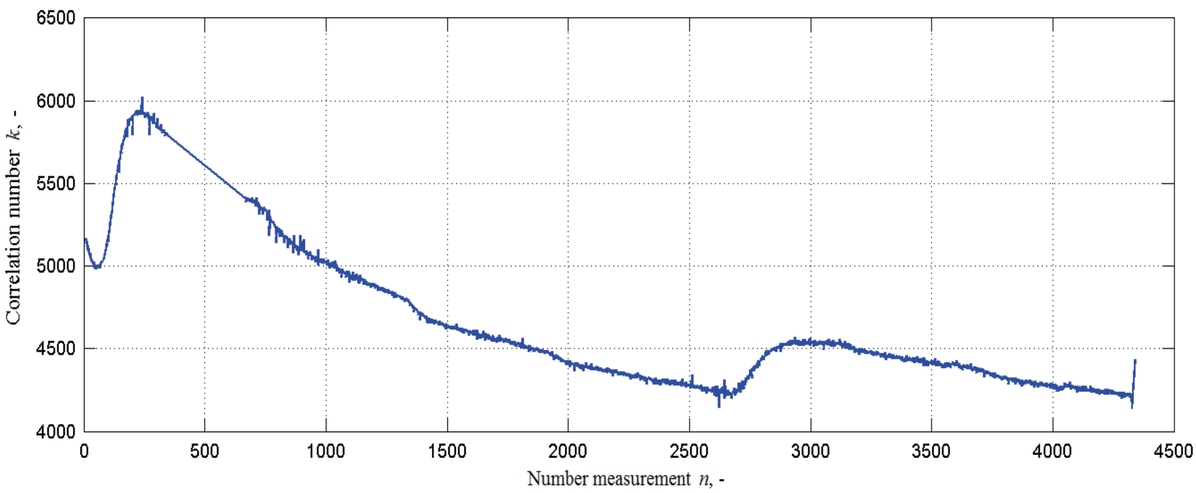


Figure 14a Measurement results – opposite electrodes (A channel), experiment II

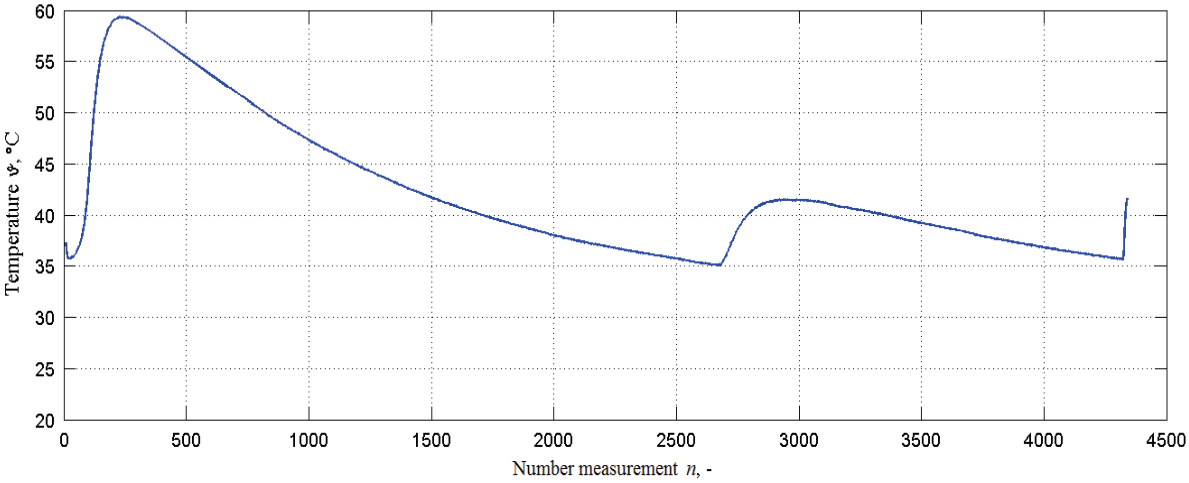


Figure 14b Measurement results – temperature change, experiment II

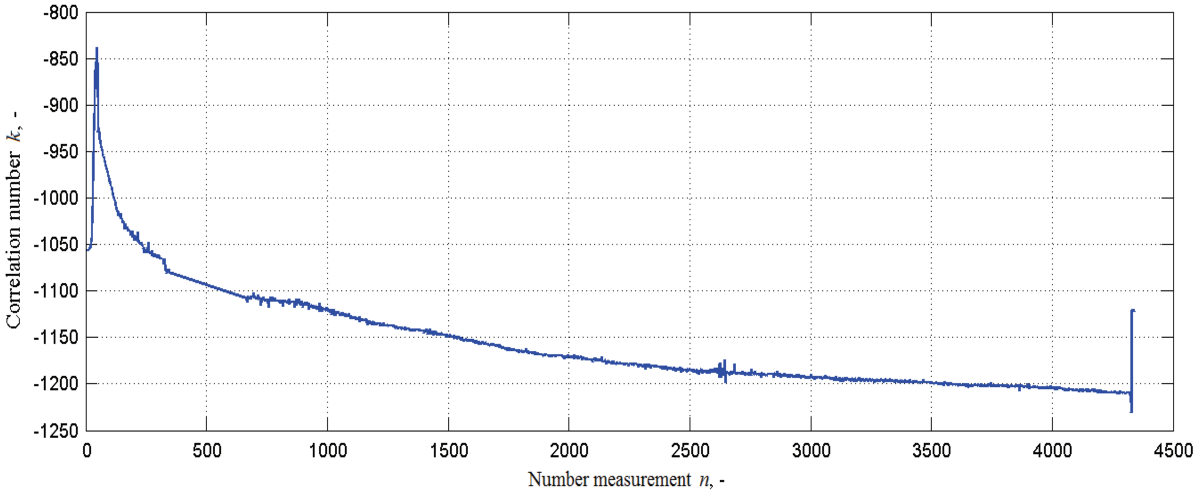


Figure 14c Measurement results – interdigital sensor (B channel), experiment II

During the third experimental measuring from the initial mass 510 g of water was evaporated from blueberry fruits placed in a layer onto a horizontal pan. Quantified values expressed by the correlation number *k* for the number of measurement *n* by using the interdigital sensor (A channel) are shown in Fig. 15a. Apart from the initial fluctuation due to the fact that vacuum was suddenly reached, moisture decrease of blueberry fruits was measured by the used sensor until the end of the drying process. The measured values obtained by using the interdigital sensor (B channel) oscillate to a substantial degree and are practically not usable (Fig. 15b). The measured temperature values during the third experimental measuring are shown in Fig. 15c.

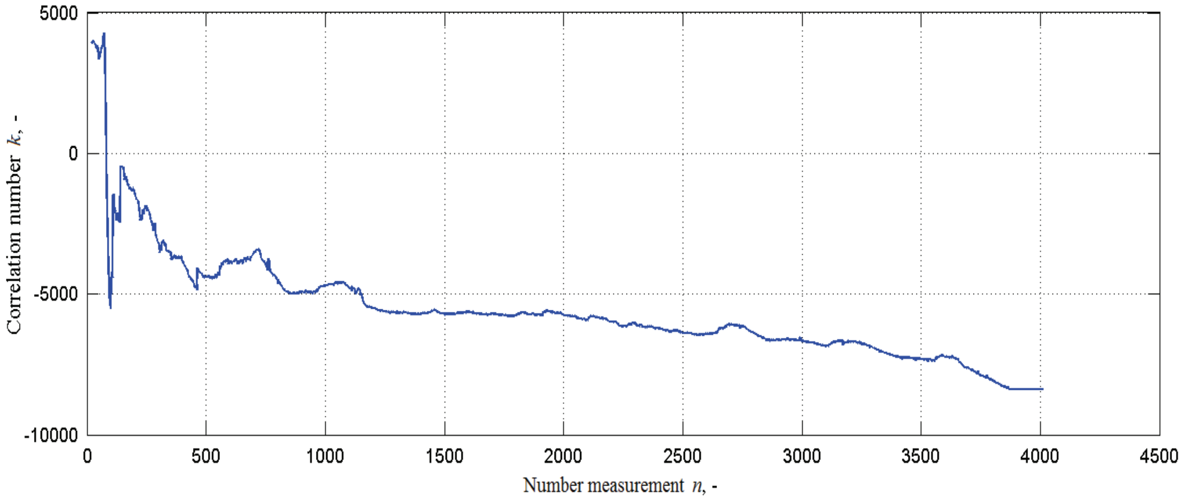


Figure 15a Measurement results – interdigital sensor (A channel), experiment III

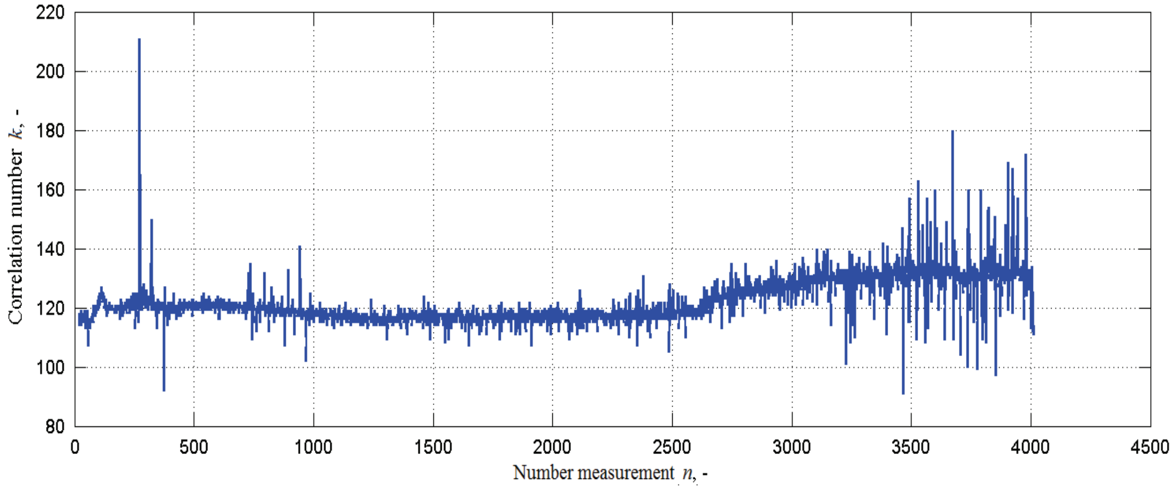


Figure 15b Measurement results – interdigital sensor (B channel), experiment III

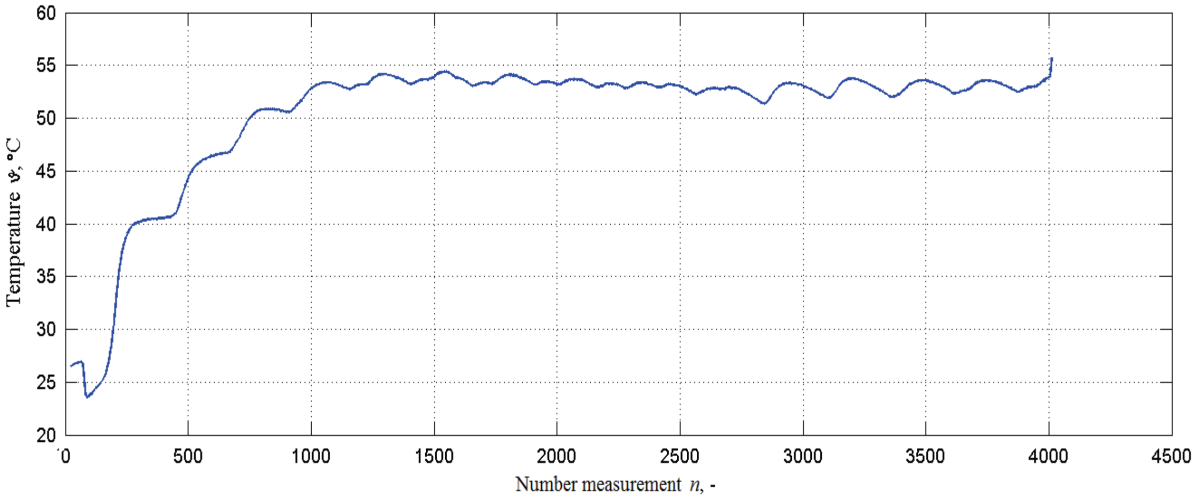


Figure 15c Measurement results – temperature change, experiment III

4 Results analysis

During the experiment the measuring device operated reliably and carried out the forseen functions. Measurements and data transfer reflected experimental conditions and moisture reduction of blueberry fruits dried by means of radiation in a vacuum chamber. During the first experiment the sensor with opposite electrodes registered moisture reduction of blueberry fruits, but the measured values (correlation number k) significantly depend on the recorded vacuum oscillations and, thereby also on the temperature change inside the chamber.

In the second experiment applicable results were achieved by using the sensor with opposite electrodes with the difference of 1883 units between the highest and the lowest value (Fig. 14a). The impact of a momentary increase in the blueberry fruits temperature on the measurement of their moisture (around 2680th measurement) is expressed due to a sudden switch-on of electric plate heaters. Interdigital sensor is almost insensitive to temperature change, and the measured values of capacitance expressed by the correlation number within the range of 393 units correspond to moisture reduction of blueberry fruits dried in a vacuum chamber (Fig. 14c).

Values measured by means of the interdigital sensor placed onto a blueberry layer that is up to 35 mm thick reflect the moisture reduction of fruits with a wider range of the correlation number k within ≈ 8000 units. The oscillation of measured values during moisture reduction of blueberry fruits is affected by temperature fluctuations in the vacuum chamber, Figs. 15a and 15c. Due to the blueberry layer being thin (10 ÷ 15 mm) and the influence of the pan material (stainless sheet metal) values measured by means of the interdigital sensor (B channel) are not usable, Fig. 15b. In this case, this is contributed to a somewhat greater depth of electric field penetration into the material λ through the blueberry fruits layer, so the impact of the pan on the measurement is more expressed.

5 Conclusion

The conducted experiments affirmed that measuring the capacitance change is a suitable (non-destructive) method for measuring moisture reduction of hygroscopic materials dried by radiation in a vacuum chamber. By means of an experimental test using a relatively cheap measuring device the possibility of measuring the blueberry fruits moisture reduction was confirmed. Quantified values determined practically applicable measuring sensitivity of the interdigital sensor with a measuring device during the drying process of blueberry fruits in a layer that is up to 35 mm thick in vacuum at low temperatures of the water boiling point in the material. Adjustments and regulation of working parameters during drying by radiation (subpressure, material temperature) affect the measurement precision due to the dielectric water constant change in a hygroscopic material. Further research should optimize the geometrical shape and the sensory element of the interdigital sensor according to the choice of material and the penetration depth of electric field for specific types, line-up manners and layer depth ranges of hygroscopic materials (fruit, vegetables, etc.). Furthermore, it is necessary to develop and improve mathematical models of applicable measuring with compensation for possible disturbances and external impacts that are suitable for the installation into the microcontroller program.

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Translation

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