Water content measurement of thin sheet starch products using a conductance technique

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Abstract

An electrical conductance technique for water content measurements of drum-drying and extrusion cooking starch products is described in this paper. It is based on the conductance measurement of a thin sheet of material between two electrically isolated metallic plates, acting as electrodes. Water content measurements are calibrated against the conventional loss-in-weight method. Once calibrated the conductance technique yields satisfactory accuracy, repeatability and stability compared to the loss-in-weight method. Yet, it offers the distinct advantage of a major reduction in measurement time (less than a minute). The latter is of critical importance, as clearly demonstrated by moisture absorption/desorption experiments. The method is easy to use and calibrate, readily installed and can be implemented at relatively low cost. The conductometric technique employed in this work, enabled rapid and reliable water content measurements of maize starch sheets (thickness of 0.085, 0.5 and 1 mm) ranging from around 0% to 20%. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Electrical conductance; Maize starch; Water content; Moisture; Drum drying; Extrusion cooking

1. Introduction

Drying plays an essential role in food technology and is subject to a fast growing interest due to increasing requirements in product quality. In order to better control and optimize the drying processes, moisture content of a food article is considered to be a key factor (Bonazzi et al., 1996). In principle, moisture determination can be regarded as a simple analytical procedure. However, such a procedure is actually beset with many complexities and the methods proposed for the moisture estimation are rarely absolute (Keey, 1972). From an economic point of view, the need for a reliable and rapid moisture content estimation is of major importance.

The most widely recognized methods for moisture determination are the oven methods, based on calculating the moisture content of the sample by the loss of its weight upon drying at a controlled temperature. These methods, though simple and direct, are time consuming, since the heating period can last several hours (e.g., Henderson, Perry & Young, 1997; Keey,

1972). Therefore, the loss-in-weight techniques are practically unsuitable for automatic control purposes and as underlined by Carr-Brion (1986) there are no industrial applications of these methods.

Nevertheless, the loss-in-weight techniques usually constitute the basic method against which other faster and more practical indirect techniques are calibrated. Quite a few indirect techniques, i.e., electrical conductance and capacitance, infrared thermometry, optical absorption, microwave attenuation, have been employed in the past with considerable success for measuring moisture in various food articles (e.g., Troller, 1983; Goldstein, Bein, Gersh, Lee & Zakin, 1991; Brzezinski & Palka, 1994; Rodriguez, Vasseur & Courtois, 1996; Zanoni, Peri, Giovanelli & Pagliarini, 1999; etc.). Several comprehensive reviews, e.g. Henderson et al. (1997), Keey (1972) and Carr-Brion (1986), outline such methods with emphasis on their advantages and disadvantages. In addition to the fact that these techniques often require a rather tedious calibration procedure, most of them are quite expensive. Specifically for the on-line moisture determination of solid products, Carr-Brion (1986) considers electrical measurements as the most attractive option for it combines effectiveness with low cost. However, it is recognized that in these

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measurements bulk density, temperature and chemical composition may play a role, so a meticulous calibration must be employed.

Drum-drying and extrusion cooking are the two main commercial thermal treatments of starchy food products, i.e., cereals, tuber and legume seeds, flours (Collona, Buleo & Mercier, 1987). Extrusion cooking is also used to produce thermoplastic starch materials, which exhibit high stability and particular functional/mechanical properties and which make them suitable as protective coatings and biodegradable packaging (Belitz & Grosch, 1987). Both drum-drying and extrusion cooking result in specific physicochemical modifications of the starch according to the severity of the treatment due to the breakdown of the starch granules (Mercier, 1987). In particular, the physical modifications in extrusion cooking, are due not only to gelatinization (hydrolysis upon heating), but also to a macromolecular degradation of both amylose and amylopectin resulting from the shearing action involved. In both processes, the delivered starch product is usually in the form of thin solid sheets of varying thickness. Customarily, drum-drying products have a thickness well beyond 1 mm, whereas extrusion cooking products are of the order of a millimeter. Industry frequently encounters problems in both technologies as regards the output product quality because of perturbations in moisture content. For a real automatic control of these processes, an on-line moisture measurement is needed. Motivated by this, Rodriguez et al. (1996) developed a technique for moisture determination of a drum-dried product, based on the in situ temperature measurement of the material by means of an infrared thermometer. The moisture content of the product was directly related to temperature via a desorption isobar. The latter was obtained using a rather complex method described in a paper by Bassal and Vasseur (1992), involving a mathematical sorption model (Oswin model) and a non-linear optimization method to calculate the parameters of the model.

In view of the above, the need for an alternative technique able to provide fast as well as accurate moisture measurements, is obvious. In addition, a simple and low cost measuring method is also demanded. The development of an improved electrical conductance method, which will eliminate some of the previous methods' disadvantages, appears to be a tempting option. Brzezinski and Palka (1994), communicated that they effectively applied an alternating current (a.c.) conductance technique to measure the water content of potato starch powder in both a laboratory and an industrial scale. Those authors described a specific instrumentation design, which was capable of successfully estimating potato starch powder moisture in the range 15–25%. However, they presented very few results to support their findings nor they gave estimated values of the parameters of the empirical equations they produced.

The scope of this work was to explore a novel conductance technique for measuring the cross-sectional averaged moisture in maize starch in the form of thin sheets leaving the process equipment. A modified version of an a.c. conductance technique originally developed for studying thin liquid films (e.g. Andritsos & Hanratty, 1987; Karapantsios, Paras & Karabelas, 1989; Vlachos, Paras & Karabelas, 1997) was employed after the appropriate alterations and tuning in the electronic circuits. In designing the experimental system, a special effort was made to broaden the range of reliable moisture measurements. The achieved useful moisture range was approximately from 0% to 20%. The role of the product texture and porous structure, arising from the processing, in influencing the moisture distribution within the material was discussed.

2. Materials and methods

2.1. Materials and processing

Commercial native maize starch was purchased from GROUP AMYLUM S.A., Greece, with a moisture content of 13.5%. The mean diameter of the granules was 14.95 μm as determined by a laser diffraction particle size analyzer (Malvern Mastersizer, Malvern Instruments). The total amylose content was $26 \pm 0.3\%$, determined by the method of Morrison and Laignelet (1983). In addition, some chemically modified maize starch – a starch-g-poly(methyl acrylate) – was donated by GROUP AMYLUM S.A., Greece. The liquid used was tap water, filtered mechanically to remove suspended particles larger than 2 μm. Its specific conductivity varied between approximately 600 and 700 μS/cm.

Native maize starch was modified by drum-drying on a double drum dryer (GOUDA). The drums had a 0.5 m diameter and 0.5 m length, and were internally heated by steam at a pressure of 6 bars. The drums were synchronously driven at a rotational speed which was varied over the range of 2–6 rpm, while the free surface of the liquid pool between the two cylinders was kept at 18 cm above the nip (0.9 mm gap setting at ambient conditions). Starch/water suspensions with solids concentration of 10% w/w were employed as the dryer feed.

Some starch/water/glycerol mixtures were thermally treated by a semi-industrial twin-screw extruder (BC 45, CLEXTRAL S.A.). The mixture of glycerol and water is customary employed in extrusion cooking as a plasticizer to furnish certain functional properties to the thermoplastic starch product (Collona et al., 1987). The solid feed to the extruder was either the chemically modified starch or the physically modified (pregelatinized) drum-dried starch produced in our lab. In both chemical and physical modification processes, the hydrophilic-hydrophobic character of the starch granules

drastically changes resulting in a variety of product textures and porous structures, e.g. Belitz and Grosch (1987). The throughput rate for the solids was 11.27 kg/h, whereas for the liquid phase was 5.25 l/h (30% water, 70% glycerol). The barrel temperature was kept at 100°C for the physically modified starch and 140°C for the chemically modified starch. The screw speed was 40 rpm at all times.

The drum-dried and the extruded products were obtained in the form of thin sheets from which samples were taken to measure their water content. The sheets produced by the drum-dryer had an average thickness of 0.085 mm, over the limited range of rotational speeds employed. However, moderate fluctuations around this value were persistently observed as a result of perturbations in the drying process. Such phenomena are often encountered in drum-drying and are responsible for producing unevenness in the moisture distribution of the final dried product, e.g. Rodriguez et al. (1996). The sheets produced by the extruder had a thickness of 0.5, 1 or 2.5 mm according to the particular die used every time. Just after production, the extruded sheets were stored at 22°C and 55% RH for 12 days in order to stabilize the material (post-crystallization).

Both drum-drying and extrusion cooking are capable of imparting a variety of textures and porous structures to the product – dictated strongly by the intensity of the treatment-which are expected to influence its water retention capacity. It is noteworthy, that the particular graft copolymer – starch-g-poly(methyl acrylate) – employed here yielded leathery translucent plastics upon extrusion. To grade the surface roughness and morphology of the sheet products, optical microscopy and stereomicroscopy were employed. Indirect gross information regarding bulk porosity was inferred from wettability tests by application of Congo red dye to stain swollen particles and further inspection of the hydration pattern and rate.

2.2. Measurement technique

A schematic of the measuring set-up is shown in Fig. 1. Electrical conductance measurements were made by means of two metallic plate electrodes. The material of the electrodes was chosen to be stainless steel so as to withstand possible wear and corrosion and to meet the strict hygienic and safety regulations for food handling. The plate electrodes measure 8.5 × 12 cm, thus providing a relatively large sensing area, which rendered the measuring method independent of local irregularities in the moisture distribution across the material. This type of dependence is considered to be a major shortcoming inherent in electric moisture meters (Henderson et al., 1997) since their readings always reflect the path of least resistance. Criteria for the selection of the surface area of the electrodes were based on the volume-averaging
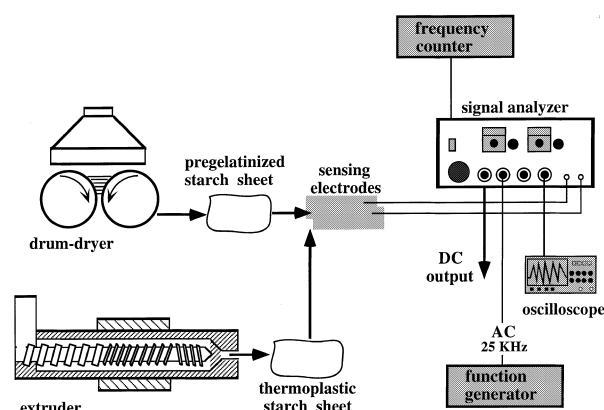

Fig. 1. Schematic representation of the measuring set-up.

approach of modeling porous media (e.g. Carbonell & Whitaker, 1984). Furthermore, the size of the present electrodes ensures reliable cross-sectional averaged measurements as it is also much larger than any spatial inhomogeneity ever noticed at the samples.

The more moist the sample the more path for electric current flow, and hence the higher the conductance. However, the surface moisture of the sample may have a great effect on the readings (Henderson et al., 1997), so measurements cannot be made when the outer surface of the material is saturated with water. Such behavior was observed in this study only with drum-dried samples having moisture well above 20%.

A piece of the starch sheet leaving the process equipment, able to cover the surface of the probe in excess, was cut and then placed in contact between the two electrodes, which were electrically isolated. In order to eliminate changes in contact resistance and ensure the repeatability of data, all the samples were pressed before measurements by a piston weight, spanning the entire electrode area, for about 30 s at a constant pressure of 5 kPa. Tests showed that pressing the samples with such a pressure, it only took about 20 s to reach a constant output signal from the measuring device, which was kept stable for at least 5 min. When pressures of 10 kPa were tested over drum-dried samples having moisture above ∼15%, the water of the sample appeared to redistribute over the material surface forming interconnecting patches across the measuring area. Higher pressures were even capable of disintegrating the least moist products (<8%). Thus, the application of a thrust of several hundreds, or even thousands, of kPa as is usually the case with starch grains (e.g. Keey, 1972), is completely unsuitable for the delicate drum-dried starch sheets. Less critical was the case with the extruded sheets. Yet, the thinner ones (0.5 mm) were also not robust enough to withstand high pressures. Altogether, measurements lasted less than a minute (including sample placement between the electrodes and pressing with the weight). The temperature of all samples during

the tests was 22°C (±1°C), as measured with an infrared thermometer.

The sensing electrodes were part of an electronic circuit, comprised of a custom-made analyzer-demodulator and a function generator. An a.c. carrier voltage of 0.1 V (rms value) was applied across the electrode pair at a frequency of 25 kHz in order to avoid electrode polarization and eliminate capacitive impedance. At this frequency, the phase angle shift between the applied voltage and the measured current across the material was virtually zero. The largest changes in conductance with moisture content occur when "bound" water is present, since "free" water tends to dampen the response (Keey, 1972). Thus, such high frequency electrical measurements enhance the sensitivity response of free water in the intergranular spaces and in the pores of the material, since strongly bound water has a resonance at much lower frequencies (Hardman, 1985).

The signal of the conductance probe was fed to the electronic analyzer, which was similar to that employed by Karapantsios et al. (1989) and Vlachos et al. (1997), for liquid thickness measurements in free falling films and in two-phase gas-liquid horizontal flows, respectively. Particular attention was given to achieve a satisfactory sensitivity over an extended moisture range in order to cope with the demands of the present study. For this, a special demodulation circuitry was included in the analyzer. This circuit acted to demodulate the peaks of the output signal synchronously using the source oscillator to create square pulses of the same frequency as the reference. A two stage cascade phase adjustment allowed the pulses to scroll about and intersect the carrier signal at any point along the cycle (where the signal from the probe was desired to be sampled), thus converting it to an appropriate analog d.c. voltage signal. The user-selected amplitude of each cycle of the output was stored on a capacitor and fed to an output terminal through a buffer amplifier. The analog voltage output of the analyzer (V) was converted to equivalent conductance of the medium between the electrodes ($1/R$) by means of a calibration curve based on precision resistors. This calibration was performed prior to and after each series of measurements, to account for variations in room temperature and input voltage. The best calibration scheme was provided by a fifth-order polynomial equation fit to the curve $\log_{10}(1/R)$ vs V. A fifth-order polynomial equation was customarily employed by Andritsos and Hanratty (1987), in order to describe their analyzer calibration curve.

Thus, by means of this equation, the analyzer's output voltages were uniquely assigned to equivalent conductance values. The signal from the electronic unit was monitored by a digital voltmeter, whereas the high frequency carrier and pulse signals by a dual band oscilloscope.

A substantial number of maize starch samples was used each time in order to establish the relation between equivalent conductance and water content. The former was computed by the voltage output of the analyzer (as described above), while the latter was determined by drying the samples to constant weight in an oven at 105°C. Finally, the following regression equation was used to correlate the sample's equivalent conductance ($1/R$) to its water content (M):

$$\log_{10}(1/R) = c_1 + c_2 M, \tag{1}$$

where c_1 and c_2 are the correlation coefficients numerically obtained. The same expression was also used by Brzezinski and Palka (1994) to determine the moisture of potato starch powders.

The conductance signal across the probes depends both on the conductivity of the water, which may vary due to temperature and ionic strength, and on the chemical composition of the starch source – especially the concentration of ionic conductors such as salts. Such a complex dependence was also observed by Brzezinski and Palka and was compensated by rationing all measurements to some reference conditions. A separate measurement of the conductivity of starch/water suspensions was proposed as a direct method to determine the appropriate reduction values and this procedure was also employed in this study. Thus, a new calibration was made every time that the material or the experimental conditions were changed. However, the significance of the varying textures of the product with respect to the moisture distribution inside the material was still a matter of concern.

3. Results and discussion

To assess the validity of the conductometric technique for the drum-dryer products, two sets of 30 drum-dried samples from the same maize starch source were used. Both sets consisted of three independent series of experiments, each experiment including at least three measurements at the particular rotational speed. The final selection of the data was based on a systematic scrutiny of experimental errors and further rejection of extreme outliers. In the first set the encountered rotational speeds were 2, 3, 4, 5 and 6 rpm, whereas in the second set were 2, 4, 5, 5.5 and 6 rpm, thus giving emphasis to the higher moisture samples. For the specific operational conditions of the drum dryer employed in this study the product sheets had water content ranging from ~3% to 20%.

The equivalent conductance of the starch samples was determined by means of the respective analyzer calibration curve. Recall that a separate analyzer calibration curve was determined for every series of experiments. The water content of each sample was

independently determined using the loss-in-weight technique. Then, a semi-implicit procedure, similar to that used by Karapantsios et al. (1989), was employed to check whether the conductance technique could accurately describe the moisture measurements. It was based on parity plots of experimental versus estimated values produced as follows: the first set of samples was employed for the calculation of the coefficients c_1 and c_2 in Eq. (1) and these values were used thereafter to estimate the water content (M) of the second set of samples. Fig. 2 displays these estimated M values against the directly determined values by the loss-in-weight technique. Over the range of measurements considered, the conductance and the loss-in-weight techniques showed a satisfactory agreement (within 10% relative). Next, the same procedure was followed, using this time the second set of samples to calculate the correlation coefficients in Eq. (1), wherefrom the water content of the first set of samples was determined. In both cases, the obtained regression equations almost coincided, as shown in Fig. 3. The noise-to-signal ratio for the electrical measurements was always below 0.01. Thus, the scatter observed in Figs. 2 and 3 reflects chiefly the fair accuracy in moisture determination by the loss-in-weight technique as a result of the small weight of the samples (having the size of the electrodes). This problem was particularly acute with the low moisture samples. The average variance V ($=$ S.D./mean) calculated from experimental values over the entire range of water content (M) was 0.06.

In order to produce samples of different water content from the sheets extruded from chemically modified starch (moisture after stabilization 9–10%), a stepwise procedure was followed. First, the conductance of the as
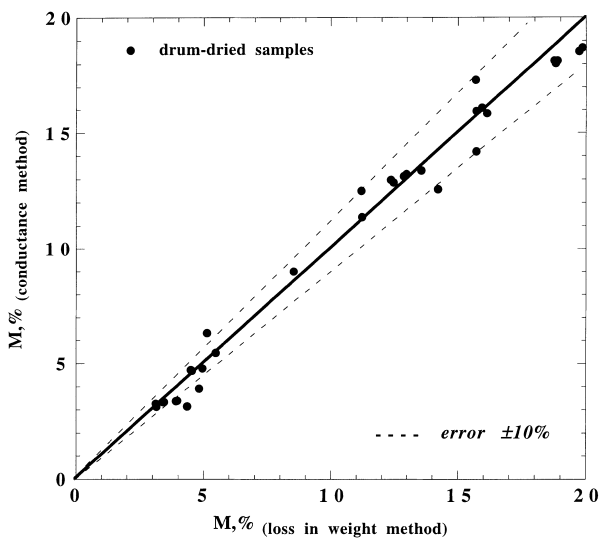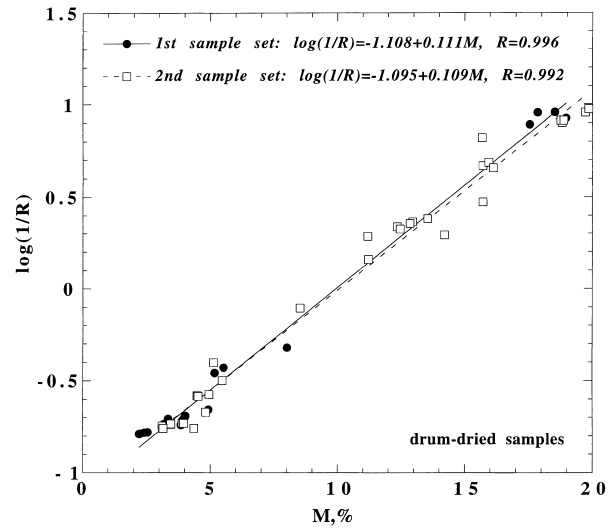

Fig. 3. Equivalent conductance of drum-dried maize starch samples correlated with their water content.

delivered from storage samples was measured. Then, the samples underwent complete drying to constant weight in an oven (105°C) and their conductance was subsequently measured. Finally, the samples were put to rehydrate inside an environmental chamber held at 22°C and 90% RH until they reached a moisture content of 17–18%. The thicknesses of these extruded starch sheets were 0.5 and 1 mm. Fig. 4 presents the corresponding regression equations for the extruded starch samples determined by the analysis described above. The different slopes of the $\log(1/R) = f(M)$ functions are attributed to the different thickness of the samples since the material characteristics were more or less the same as a result of the constant processing conditions.

Fig. 2. Parity plot of starch water content values measured using the conductance method versus those measured by the loss-in-weight technique.
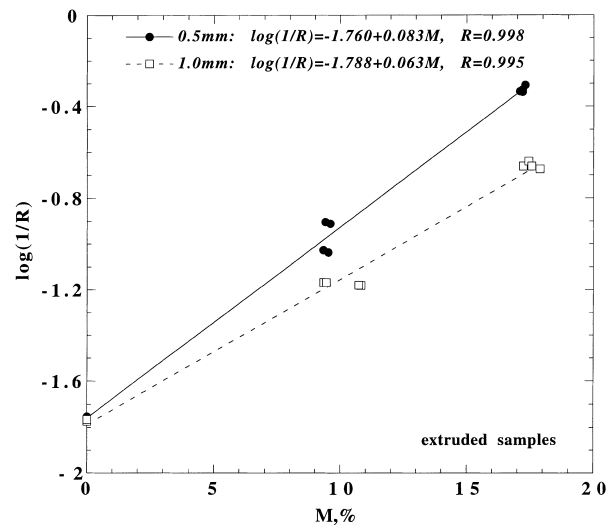

Fig. 4. Equivalent conductance of extruded maize starch samples correlated with their water content.

The obtained drum-dried sheets present excellent wettability and are easy to rehydrate, qualities very important for ready-to-use products (e.g. Bonazzi et al., 1996; Rodriguez et al., 1996). However, their affinity to moisture makes the automatic control of drum-drying equipment very cumbersome due to the short response time required for an on-line moisture measurement. To demonstrate the importance of obtaining rapid moisture measurements, humidity absorption/desorption tests were carried out in our lab. The samples were placed on a sensitive balance standing inside an environmental chamber which was set at 22°C and 55% relative humidity. Three separate runs were conducted at all experimental conditions to check for repeatability. Fig. 5 depicts such absorption/desorption experiments with drum-dried sheets obtained at different rotational speeds (2–7 rpm). Samples treated at high rotational speeds (6 and 7 rpm) had higher initial water content and appeared to rapidly desorbe moisture towards an equilibrium stage. On the contrary, the initial water content of samples treated at low speeds (2 and 4 rpm) rapidly increased by absorption towards equilibrium. In all cases, few minutes were just enough for sample moisture to change drastically. The equilibrium stage appeared to be around the moisture value of the purchased commercial maize starch (13.5%).

To check further on the material characteristics upon water uptake, some absorption tests were conducted with drum-dried samples completely dried first (to zero moisture) and then placed inside the environmental chamber at 22°C and 55% RH. The balance readings taken from time to time are shown in Fig. 6. In all runs, the absorption curves appeared to have the same overall pattern although the scatter within the main path of the

Fig. 6. Absorption curves of initially dried drum-dried starch sheets produced at various drum rotational speeds.

curves was appreciable (variance 0.1), with no obvious dependence on rotational speed. One can further see that the moisture content increased markedly with time (towards an equilibrium stage). The equilibrium stage was initially approached at a considerably faster rate than in the experiments of Fig. 5. The resemblance among the absorption curves of Fig. 6 is remarkable especially if one considers the variations observed in the texture and porosity of the samples treated at different rotational speeds. The drier the samples the more surface uneven and easier to rehydrate. Thus, one might be tempted to argue that, within the range considered, differences in material properties may not seriously affect the response of the conductance probe.

Moisture absorption experiments were also carried out with sheets extruded from both chemically and physically modified starch. The former had 0.5 and 1 mm thickness, whereas the thickness of the latter was 2.5 mm. Again, the samples were dried to constant weight before being placed inside the environmental chamber. Note that sheets from physically modified starch had a much higher equilibrium moisture of 18–19% (after stabilization) than sheets from chemically modified starch (9–10%). The results are shown in Fig. 7. Clearly, with extruded sheets moisture uptake was not as rapid as with drum-dried samples. Different absorption rates were measured with sheets produced from chemically modified starch having different thickness. The thicker the samples the slower the moisture uptake.

The largest effect though, was that due to the type of the modified starch used. Sheets produced from physically modified starch appeared to have a significant water gain rate despite their thickness. At short times, this rate was comparable to that of sheets produced from chemically modified starch with 0.5 mm thickness,
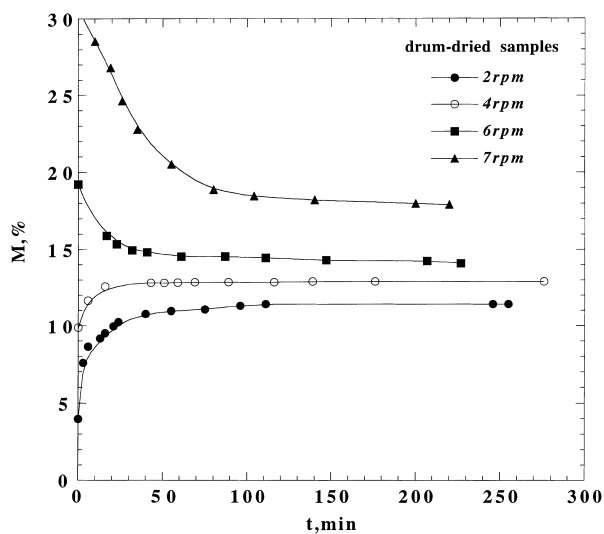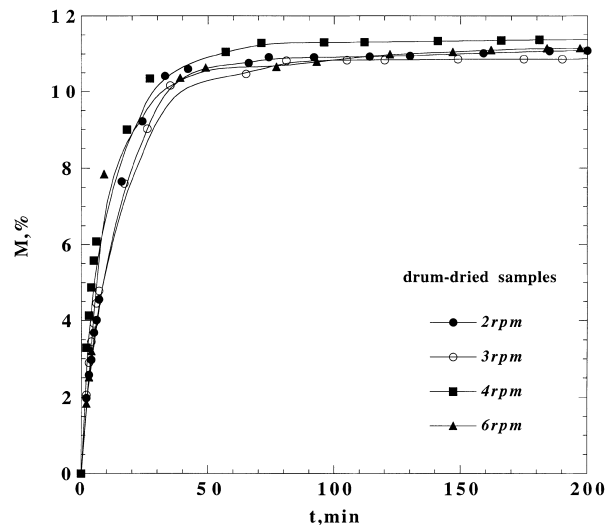

Fig. 5. Absorption and desorption curves of as-sampled drum-dried starch sheets with initial water content, produced at various drum rotational speeds.
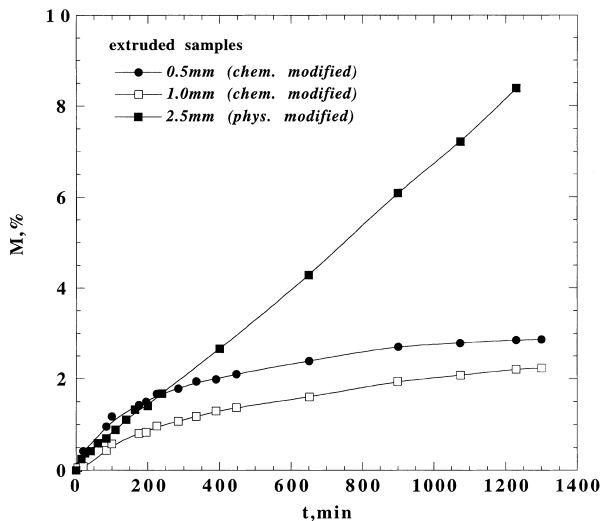
Fig. 7. Absorption curves of initially dried extruded starch sheets.

but accelerated substantially at later times. At least part of this behavior may be attributed to the higher equilibrium moisture of the sheets produced from physically modified starch. This observation, if verified by further studies, indicates that thermoplastic starches produced from physically modified samples may have a faster adjustability to variations in external conditions than products from chemically modified starches. In this case, a rapid moisture estimation of the product sheets leaving process equipment is needed such as the one provided by the present conductometric technique. The time needed for water content measurements was (for all tests) less than a minute, since no special sample preparation was required and the pressing of the sample between the sensing electrodes was just 30 s.

4. Conclusions

An electrical conductance technique was used to effectively measure the moisture of thin maize starch sheets leaving drum-drying or extrusion cooking equipment. The technique presented satisfactory overall accuracy ($\pm 10\%$ relative) on measuring water content in the range from around 0% to 20%. Advantages of the present electrical conductance technique, over other traditional methods, include its low cost as well as its ease of installation, calibration and operation. Moreover, as shown by moisture absorption/desorption tests carried out in our laboratory, time may be of critical importance for water content determination in process equipment. The conductance probe used in this study provided fast and dependable measurements; samples were analyzed in less than a minute with no special sample preparation.

In order to apply the method for actual on-line moisture measurements, in an industrial scale drum-dryer, additional work is already underway in our lab. The main problem is that of establishing good contact at all times between the electrodes and the product sheet. Preliminary tests in which the drum-dryer's knife was utilized as a part of the conducting electrode pair, appeared to have the potential to solve this problem.

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