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Innovative application of the moisture analyzer for determination of dry mass content of processed cheese

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Abstract

The aim of this work was the presentation of an alternative method of determination of the total dry mass content in processed cheese. The authors claim that the presented method can be used in industry's quality control laboratories for routine testing and for quick in-process control. For the test purposes both reference method of determination of dry mass in processed cheese and moisture analyzer method were used. The tests were carried out for three different kinds of processed cheese. In accordance with the reference method, the sample was placed on a layer of silica sand and dried at the temperature of 102 °C for about 4 h. The moisture analyzer test required method validation, with regard to drying temperature range and mass of the analyzed sample. Optimum drying temperature of 110 °C was determined experimentally. For Hochland cream processed cheese sample, the total dry mass content, obtained using the reference method, was 38.92%, whereas using the moisture analyzer method, it was 38.74%. An average analysis time in case of the moisture analyzer method was 9 min. For the sample of processed cheese with tomatoes, the reference method result was 40.37%, and the alternative method result was 40.67%. For the sample of cream processed cheese with garlic the reference method gave value of 36.88%, and the alternative method, of 37.02%. An average time of those determinations was 16 min. Obtained results confirmed that use of moisture analyzer is effective. Compliant values of dry mass content were obtained for both of the used methods. According to the authors, the fact that the measurement took incomparably less time for moisture analyzer method, is a key criterion of in-process control and final quality control method selection.

1 Introduction

Processed cheese is generally formed by blending of natural cheeses with melting salts and water using heat and agitation [1, 2]. According to authors [3] processed cheese can be made from a simple basic component such as cheese, water and melting salts as well as from a complex mix of components with various types of proteins, fats, gums, stabilizers, flavours, humectants and added minerals. From physical point of view processed cheese is an oil-in-water emulsion. Water in

processed cheeses has a role to produce a smooth and stable emulsion [2]. Water, according to its unique properties, dissolves the calcium chelating salts as well as hydrates the proteins and disperses the remaining ingredients. To achieve desirable texture properties of a product such as spreadability or meltability in processed cheese slices water is necessary. Moreover, due to the economical purposes water is added for cost reduction [2].

During processed cheese production, moisture variations can be expected due to the variability or the quality of the raw materials used and the processing conditions. Thus, the determination of moisture content is important to comply with the requirements of product specification [4]. Moisture variations can affect the textural properties and shelf-life of processed cheese [2, 5, 6]. Besides, the content of water can affect the parameters of final product such as microbiological stability or compliance with legal or industry requirements.

Due to this fact the moisture content in processed cheese must be controlled thoroughly. This parameter is tested by comparison of sample mass before and after the process of heating at the increased temperature [7]. Drying is the operation which

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allows water to be removed from the product. As a result, dry substance, defined as total dry mass content, is obtained [8]. Duration of dry mass content test in case of the reference method takes more than 4 h [9]. This can be a problematic issue, especially for the industry, where an information should be obtained within a short period of time. Such need might arise particularly during a technological process. Moisture content determination becomes a key issue when corrective actions regarding realisation of the whole process should be performed.

The time of in-process control should not be longer than a few, or over a dozen of minutes. Short time allows for instant reaction and correction of technological process parameters. In-time reaction means reduced risk of low quality product or shortened shelf-life time, which is unacceptable in food industry.

Thus, searching for an alternative method aims to provide precise, repeatable information within much shorter period of time. Develop of the new method usually requires a number of tests to be done. Unfortunately there are few tests and publications concerning the determination of water content in processed cheese, performed using reference method or other test methods. The paper presents developed by the authors methodology of water content determination, carried out by means of the moisture analyzer. Moisture analyzer allows to obtain reliable result, comparable to the one obtained using a reference method within much shorter period of time.

2 Material and methods

2.1 Material

Following commercial product were used:

- Hochland cream processed cheese, (CH1), ingredients: water, cheese (28%), skimmed milk powder, butter, emulsifying substances E450, E452, E331, milk protein, salt, acidity regulator: citric acid;
- Sertop processed cheese with tomatoes, (CH2), ingredients: cheeses, water, butter, skimmed milk powder, dried whey (from milk), emulsifying salts E450, E452, E339, dried tomato 1.7%, salt;
- SM Mlekovita processed cream cheese with garlic, (CH3), ingredients: cheese, water, butter, curd cheese, milk and dried whey, emulsifying salts (E-450, E-451), garlic (0.2%), salt, acidity regulator E-339.

2.2 Methods

2.2.1 Reference method

Total dry mass content was determined in the sample in accordance with standard guidelines [9]. Respectively, prepared samples were mixed with silica sand. Afterwards, the mixtures were dried at the temperature of 102 °C for 4 h. Blank test was carried out, a container with silica sand was dried. The test aimed to determine potential deviations of the determination being a result of drying the silica sand, which was used as a base for the tested samples. The deviations should be taken into account in the calculations. Total dry mass content was determined using the following equation:

$$w_t = \frac{(m_2 - m_0) - (m_3 - m_4)}{(m_1 - m_0)} \ x \ 100\%$$

where:

w_t – sample's dry mass content, expressed as mass fraction [%]

m_0 – mass of the container [g]

m_1 – mass of the analysed sample and container prior drying [g]

m_2 – mass of the analysed sample and container after completed drying [g]

m_3 – mass of the container used in a blank test [g]

m_4 – mass of the container prepared for drying before the drying [g]

2.2.2 Moisture analyzer method

For alternative determination of dry mass content, MA 50.3Y series moisture analyzer, manufactured by Radwag Wagi Elektroniczne, Radom, Poland, was used. Specification sheet of the moisture analyzer indicates that the reading unit for measurement is 0.001%. Drying temperature range is 40 °C - 160 °C, with 1 °C interval. Mass of a sample was analysed constantly throughout the whole heating process. Variation of mass value over the time was observed on the moisture analyzer display, it was presented as a digit value and in a graphic form (Fig. 1).

Moisture analyzer operation consists of simultaneous weighing and heating of the sample. In general this method is similar to the reference method (specified by respective standard). The weighing module for mass measurement is installed in the bottom part of the device. Over the bottom part, a drying chamber is mounted, in which the analysed sample is placed. Both these elements are coupled, which allows to weigh and heat the sample in a at the same time. On the basis of the obtained weighing results, the device calculates dry mass content of the analysed sample by the following equation:

$$\%D = \frac{m_2}{m_1} \cdot 100\%$$

where:

$\%D$ – dry mass content

m_1 – start mass of the sample
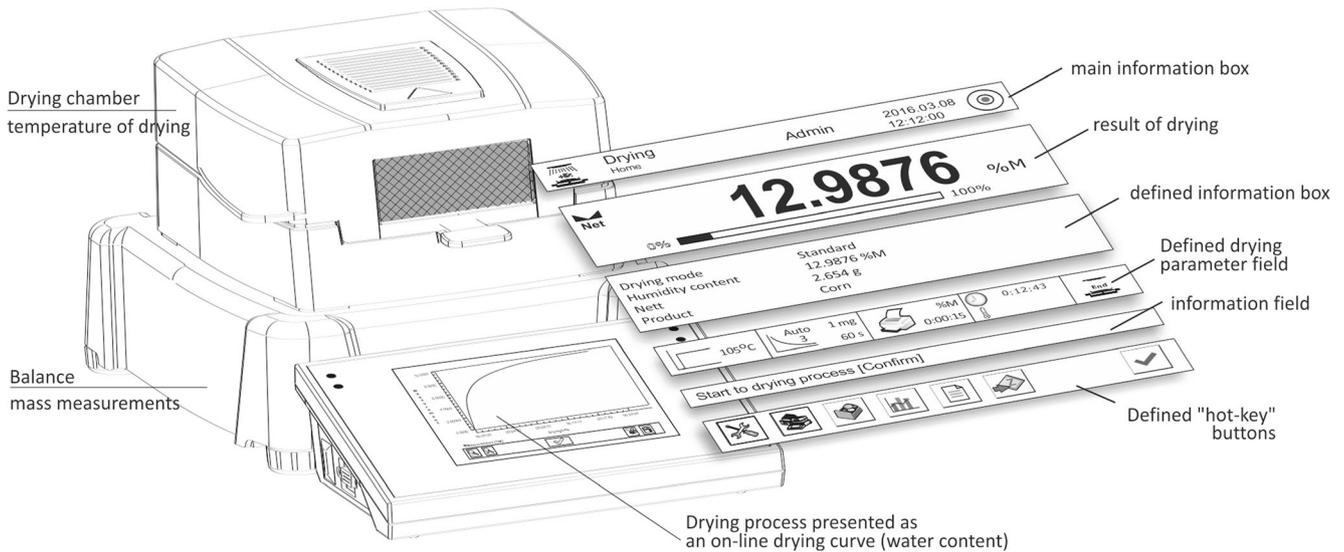
m_2 – end mass of the sample

Fig. 1 MA 50.3Y moisture analyzer

During tests the methodology was modified with regard to drying temperature and sampling. Through empirical observation, an optimal drying temperature of 110 °C was determined. A thin layer of the sample was applied onto a pre-dried aluminium pan. Next, with reference to a number of performed tests, finish mode was set, automatic 1 option was selected. Automatic 1 finish mode type means control of stability of sample mass in the amount of 1 mg over 10 s. Figure 2 presents the principle of the mechanism which controls finish mode.

The analysed sample initial mass was m_1 (Fig. 2). During the first stage of the drying process, dynamic change of initial sample mass was observed, the weigh value got reduced (curve from A to B). This is a result of a rapid increase of the drying chamber temperature. Sample mass was registered online, thus the decrease of sample weight was known during the whole drying process (Δm). Complete removal of water from the sample meant that stable final mass of the sample m_2 was obtained. The drying process ended when the mass of the

dried sample remained unchanged during one of the specified time intervals. The only changeable parameter in this process is the test duration (Δt). Through selection of time interval it is possible to influence the final mass of a sample m_2, and at the same time the value of calculated total dry mass content. The above presented method of final result interpretation allows to optimise the drying methodology. The method consists in searching of moisture analyzer parameters, which allow to obtain a result comparable to the one provided using a reference method.

2.2.3 Comparison of a reference method and a moisture analyzer method

In case of the reference method [9], the sample prior drying is mixed with silica sand. Such operation is performed in order to increase the active drying surface of the sample as well as to eliminate formation of crust on the sample surface. In this process the sample is heated by means of convection air flow in the

Fig. 2 Principle of operation of automatic finish mode. Legend: m^1- initial mass, m^2- final mass

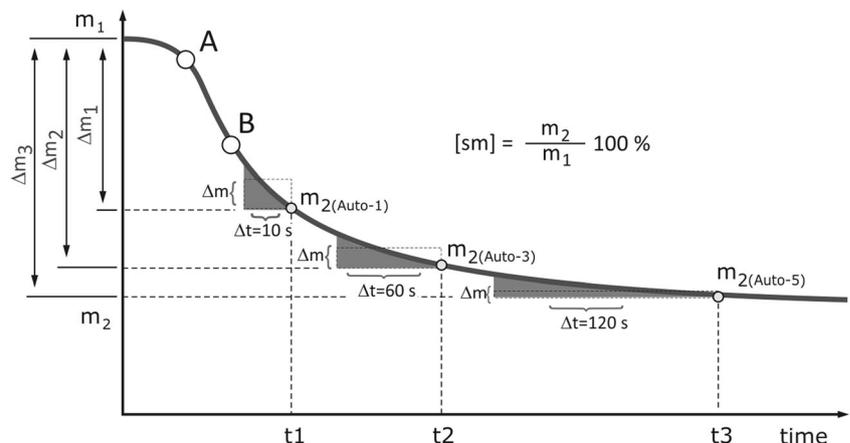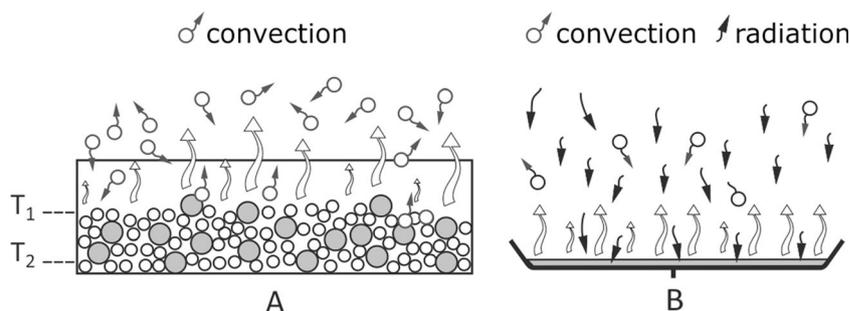$$[sm] = \frac{m_2}{m_1}\ 100\ \%$$

Fig. 3 Samples heating diagram

drying chamber (Fig. 3a). In case of convection heating, the surface layer of the sample has the highest temperature at the beginning (T1) (Fig. 3). If the sample is thick then a lot of time is required in order the entire volume (T1 = T2) reached the target temperature value, even a few hours. Removing water from the sample requires a lot of time likewise. Upon competed drying process it is necessary to additionally control stability of mass of the container with a sample. This lengthens analysis duration by extra 60 min. After this time the sample reaches the ambient temperature value, next mass measurement proceeds. When using moisture analyzer, a thin layer of the sample is spread over the weighing pan. It is heated using convection and IR radiation (Fig. 3b). Combination of these two types of heating allows to fasten the drying process [10].

In case of combined heating process (radiation and convection) the key component is infrared radiation, more precisely the wavelength. The moisture analyzer emits radiation of wavelengths ranging from 1.2 μm to about 5 μm. The wavelength depends on the installed heat source, the longer the wave, the deeper sample layers are penetrated [11].

Similarity of the reference method to the method using moisture analyzer when it comes to weighing – drying – weighing - calculation, is obvious. This is the reason why many operators use reference method parameters while operating the moisture analyzer. This is not always right. Observed deviations of the obtained results are mainly an effect of various mechanical designs of the two measuring systems, i.e. different mechanisms providing heat, mainly IR radiation of the heating lamps [10]. In practice the key issues concerning the drying process is the drying temperature, sampling and analyzed sample size (Bradley 2010). Being aware of relation between these three parameters is crucial in order to provide precise results. In case of doubts on reliability of the obtained results, validation of the drying method is required. Validation is the best solution

when reference methods are replaced with the methods based on a moisture analyzer. During moisture analyzer determination, sample mass is controlled online. There is no need to control its stability like for the reference method, which helps to significantly shorten the drying process.

2.2.4 Preliminary statistical analysis of the measurements – Hampel test

Using the moisture analyzer, a series of measurements was carried out for each of the samples in order to check the drying method repeatability. Statistical evaluation for the obtained results was performed using Hampel test in order to determine whether the data set comprises outliers.

Hampel test methodology:

1. the results were set in an ascending order,
2. M_e median value was calculated from all the results x_i, where x_i covers the range from x_1 to x_n,
3. absolute values of deviations r_i from median value were calculated for each result, calculation was done using the following equation:
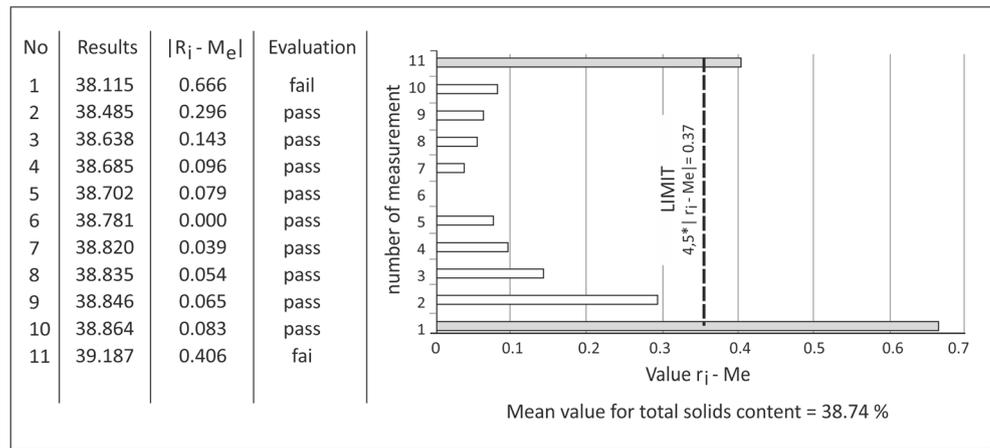
$$r_i = x_i - M_e$$

4. values $|r_i|$ were set in an ascending order, deviations median $M_e |r_i|$ was calculated,
5. the obtained $|r_i|$ values were compared with the value of 4.5 Me $|r_i|$, checking whether the following condition is met or not:

$$r_i \geq 4.5 M_e |r_i|$$

Table 1 Comparison of dry mass content results and drying time values

Sample	Total dry mass content [%]		Analysis duration [min]	
	EN ISO 5534	MA 50.3Y moisture analyzer	EN ISO 5534	MA 50.3Y moisture analyzer
CH1	38.92	38.74	240	9
CH2	40.37	40.67	240	16
CH3	36.88	37.02	240	16

Fig. 4 Statistical evaluation of dry mass content values - Hampel test for CH1

No	Results	$|R_i - M_e|$	Evaluation
1	38.115	0.666	fail
2	38.485	0.296	pass
3	38.638	0.143	pass
4	38.685	0.096	pass
5	38.702	0.079	pass
6	38.781	0.000	pass
7	38.820	0.039	pass
8	38.835	0.054	pass
9	38.846	0.065	pass
10	38.864	0.083	pass
11	39.187	0.406	fai

Mean value for total solids content = 38.74 %

If the above condition was met it meant that the measurement value is an outlier and should not be taken into account in statistical analysis.

3 Results and discussion

Most of the research is still focused on the understanding of the drying mechanisms and product quality rather than on control of the operation itself [12]. Assumption of this paper was to present a simple and alternative methodology for determination of moisture content in the course of the technological process.

Determination of dry mass in processed cheeses was carried out in accordance with reference method specified by EN ISO 5534 standard, simultaneously the same product was dried using moisture analyzer. While heating the sample using a reference method, is Due to the convection air flow that supplies the heat in reference method, due to this the process is at least 4 h long [13]. It must be stated that although this method requires a lot of time, it is a compulsory test allowing to determine reliable final result of water content of water in a product. The reference method is used in this work in order to provide a reference result, to which a result obtained using moisture analyzer method is compared. The carried out determinations showed that the total dry mass content obtained using moisture analyzer method was compliant with the result obtained using the reference method. The difference between the results ranged from 0.18% to 0.30%. Time of the experiment has been reduced considerably for all the samples. In case of CH1 sample, the measurement took 9 min, for the remaining two, CH2 and CH3–16 min (Table 1).

On a basis on performed test, the optimal value allowing to finish the drying process was control of sample mass stability over 10 s for cheese sample CH1, 25 s for cheese sample CH2 and 35 s for cheese sample CH3.

Hampel test was carried out in order to check if there are no outliers in the series of measurements. Outliers influence determination of the mean value of dry mass content, and at the same time the evaluation of accuracy of the whole drying process.

In the series of measurements performed for cheese CH1, two outliers were detected using the Hampel test. The mean value for a total dry mass content was 38.74% (Fig. 4).

Hampel test carried out for cheese CH 2 showed no outliers (Fig. 5). Mean value of total dry mass content was 40.67%.

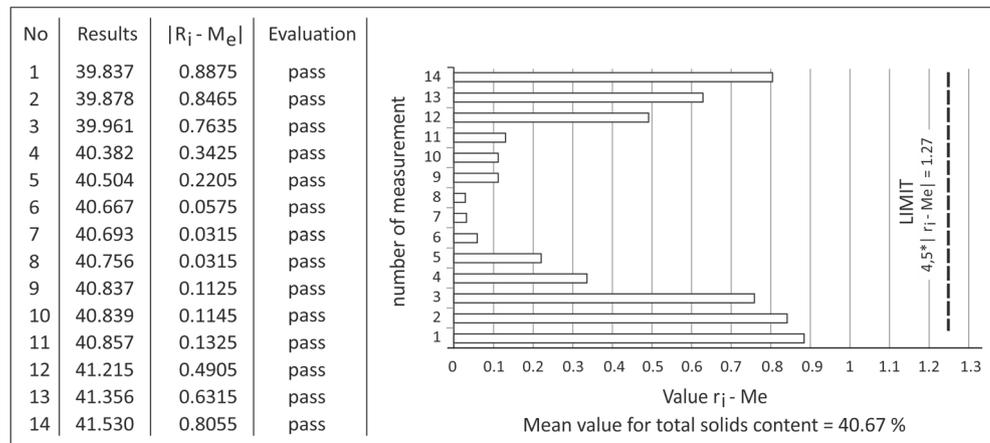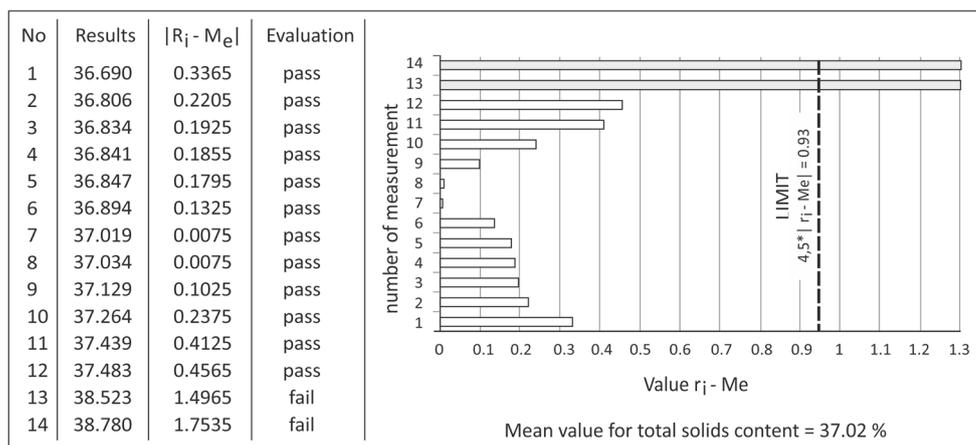**Fig. 5** Statistical evaluation of dry mass content values - Hampel test for CH2

No	Results	$|R_i - M_e|$	Evaluation
1	39.837	0.8875	pass
2	39.878	0.8465	pass
3	39.961	0.7635	pass
4	40.382	0.3425	pass
5	40.504	0.2205	pass
6	40.667	0.0575	pass
7	40.693	0.0315	pass
8	40.756	0.0315	pass
9	40.837	0.1125	pass
10	40.839	0.1145	pass
11	40.857	0.1325	pass
12	41.215	0.4905	pass
13	41.356	0.6315	pass
14	41.530	0.8055	pass

Mean value for total solids content = 40.67 %

Fig. 6 Statistical evaluation of dry mass content values - Hampel test for CH3

No	Results	$|R_i - M_e|$	Evaluation
1	36.690	0.3365	pass
2	36.806	0.2205	pass
3	36.834	0.1925	pass
4	36.841	0.1855	pass
5	36.847	0.1795	pass
6	36.894	0.1325	pass
7	37.019	0.0075	pass
8	37.034	0.0075	pass
9	37.129	0.1025	pass
10	37.264	0.2375	pass
11	37.439	0.4125	pass
12	37.483	0.4565	pass
13	38.523	1.4965	fail
14	38.780	1.7535	fail

Mean value for total solids content = 37.02 %

In the series of measurements carried out for cheese CH3, two outliers were detected using the Hampel test. The mean value for a total dry mass content was 37.02% (Fig. 6).

In order of validation of the suggested method, one of the test performed was accuracy checking. The evaluation was carried out on the basis of comparison of mean values from series of measurements with the value obtained using the reference method (Table 2).

Figures 7, 8 and 9 present graphic interpretation of the above values, where distribution of measurements from the series is shown in relation to the mean values. Figure 7 presents distribution of values of successive measurements for CH1 sample. Grey squares, in accordance with Hampel test results, are assumed to be outliers. The outliers were excluded from the statistical analysis. On the basis of the remaining measurements, a mean value was calculated. δ–1 symbol stands for difference (0.18% of total dry mass content) between the mean value of all measurements (accepted results) and the reference value. It was concluded that analyzed

sample of small size required short drying time (about 9 min). Providing analyzed sample of greater mass value, to 5 g - 7 g, resulted with longer drying time (more than 40 min). It was observed that increase of the drying temperature to over 120 °C caused surface burning of the sample. Thus, obtained results were burdened with serious errors.

The greatest variation of indications was obtained for series of measurements of CH2 sample (Fig. 5). It was noted that cheese structure was heterogeneous. It featured tiny bits of tomatoes. In general, it was observed that heterogeneous structure results with greater variation of measurements indications for unaltered test methodology. Accuracy of determination of total dry mass content in this case was 0.30% - symbol δ–2 (Fig. 8).

The highest measurement accuracy (0.14% marked as δ–3) was obtained for CH3 sample (Fig. 9). Statistical analysis excluded two outliers, specified by means of Hampel test. Similar value of measurement accuracy was obtained for CH1. It was noted that both of the samples were characterized

Table 2 Comparison of results confirming accuracy of the drying method

Sample	Reference value EN-ISO 5534 [%]	MA 50.3Y moisture analyzer [%]	Accuracy [%]
CH1	38.92	38.74	0.18
CH2	40.37	40.67	−0.30
CH3	36.88	37.02	−0.14

Fig. 7 Evaluation of method accuracy for drying with use of moisture analyzer for CH1 sample
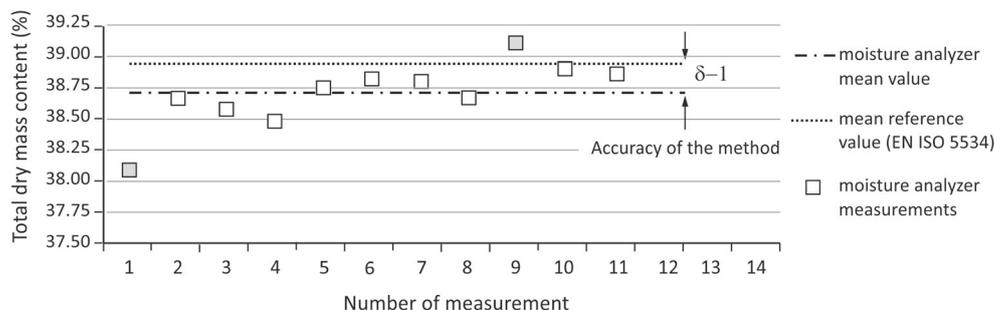
by a homogeneous structure. Homogeneity is a basic requirement allowing to obtain repeatable results of dry mass content [14].

To confirm reliability of the method, repeatability test were performed. In accordance with the standard [9], results repeatability is an absolute difference between two unrelated determination results. The measurements must be carried out in a short period of time with use of the same method by one expert with the same test material. The difference should not be greater than 0.35% [9]. The results obtained using the moisture analyzer are presented in Table 3.

Table 3 shows that for CH1 sample the obtained value exceeds the limit threshold by merely 0.03%. In case of cheese CH3 the value is exceeded by 0.45%, and for cheese CH2 by 1.35%. On a basis of obtained results evaluation, it was concluded that repeatability for the suggested method using moisture analyzer is slightly worse than for the reference method. However, the suggested method is to complete the reference method, not to replace it.

During the tests it was also checked what is the relation between the drying time and the start sample weight. In case of results obtained for CH3 sample it was observed that the relation is of linear nature. The greater the sample mass, the longer the drying time (Fig. 10).

For samples mass ranging from 2.5 g to 3.0 g, the drying process took about 16 min and the obtained dry mass content results were comparable. Samples of a greater start mass (4 g and more) lengthen the drying time to about 20 min. Greater start mass value of a sample means that thicker layer is to be

Table 3 Absolute difference between two unrelated results of the tests performed using a moisture analyzer

Sample	MA 50.3Y moisture analyzer Value |Max − Min| [%]*
CH1	0.38
CH2	1.70
CH3	0.80

*It is an absolute difference between two unrelated determination results - difference between the max and the min value of a given measurement series. The result is given as a percent value of dry mass content

dried. This can favour formation of crust over the sample surface (Fig. 11). The crust may be a result of too high drying temperature, in consequence the sample sealing occurs rapidly. Crust formation process is shown in Fig. 10 with use of a broken dotted line. The process is so too rapid that thus not all the water contained within the sample is able to evaporate [15]. Therefore water migration from inside of the sample is insufficiently intense, and the water release process may get inhibited. As a result short drying time is obtained, however accuracy of the method is very low. According to authors [16], in most drying operations, energy is transferred from the surface to the centre of the wet material with the exception of radiofrequency and microwave drying, where the energy supplied generates internal heat within the solid.

In this study, also drying times for samples of the same mass but heated using different heat sources were presented.

Fig. 8 Evaluation of method accuracy for drying with use of moisture analyzer for CH2 sample
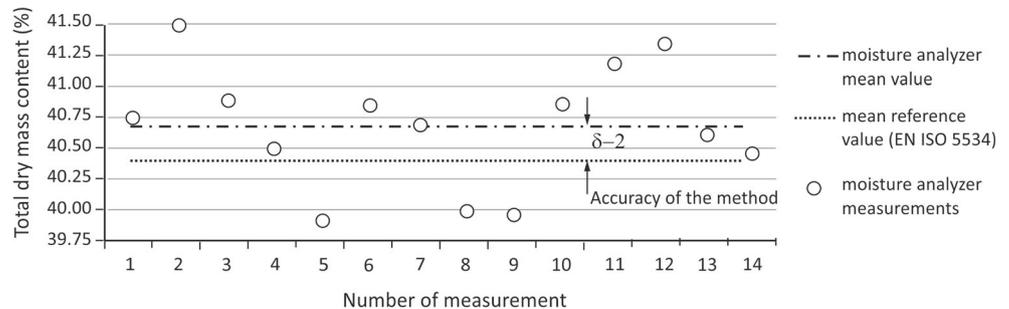

Fig. 9 Evaluation of method accuracy for drying with use of moisture analyzer for CH3 sample
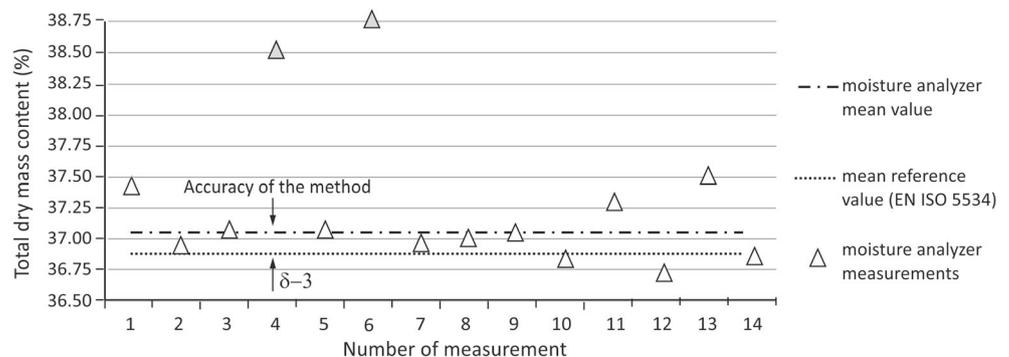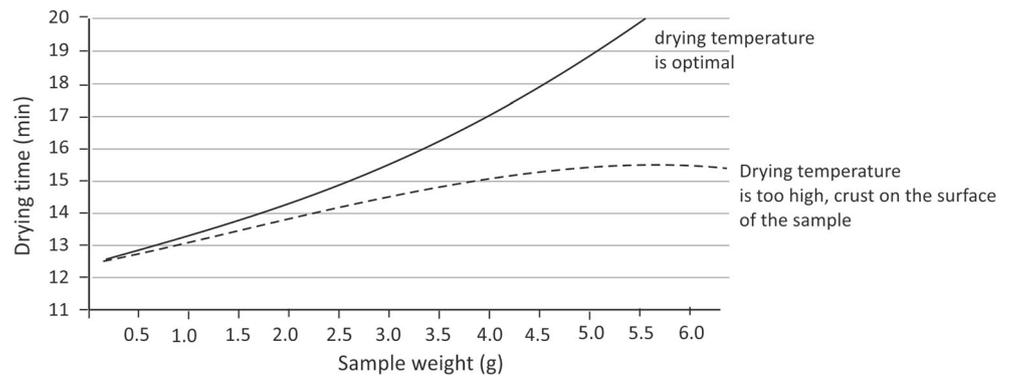
Fig. 10 Relation between the drying time and the start mass of an analytical sample

The test was carried out for an infrared emitter (IR), a steel heater (ST) and a halogen lamp (HAL). In order to eliminate potential moisture from the surface of the moisture analyzer pan, preliminary drying was performed prior the each measurement (so called pre-drying). Analysis of the drying curves lead to a conclusion that drying with use of steel heater requires the shortest period of time (Fig. 12). This is due to the fact that (ST) emits infrared radiation of the greatest wavelength. With use of this type of radiation the sample can be penetrated more efficiently [17]. When it comes to samples reaction to temperature change, the quickest response was observed in case of use of IR emitter. This can be easily noted during the first stage of the drying process (Fig. 12). The sample dried using steel heater and radiator got heated slower.

The longest drying time was obtained for a sample heated using HAL lamp. Irrespectively of the differences of cheese samples drying, the end results of total dry mass content were comparable (Table 4).

In order to show how useful moisture analyzer method is for either quality or inter operational control, rough analysis of cost has been presented. The analysis include the instrument and accessories price, economic point of view accounting for workforce cost, operation time and power consumption has also been provided (Table 5).

The analysis of cost for single determination of dry mass content for processed cheese clearly shows that the new method using moisture analyzer is more economic, more easily accessible and less demanding when speaking of creation of

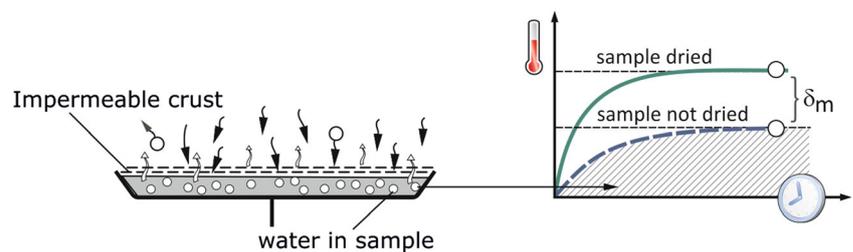**Fig. 11** Impermeable crust on the sample's layer

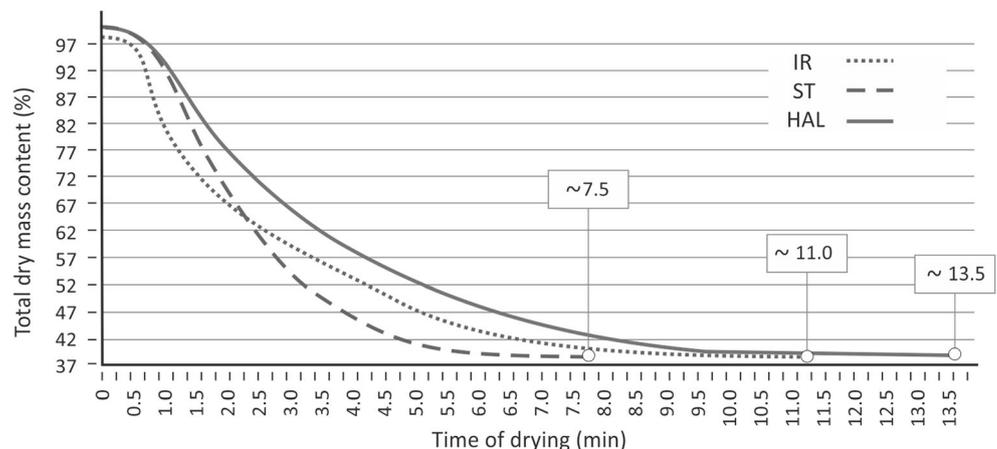**Fig. 12** Relation between the drying time rate and the heat source

Table 4 Comparison of drying results with different heat source

	IR	HAL	ST
Total dry mass content [%]	38.3	38.2	38.0
Analysis duration [min]	11.5	13.5	8.0
Sample's mass [g]	2.3	2.6	1.9

workstation for determination of moisture/dry mass content (by potential processed cheese manufacturers). Moisture analyzer may be a tool used to enable fast operational control of the final products, it may also be a response to suggestion of the authors [18] who recommend reduction of cost of the drying process. Obviously the moisture analyzer cannot be used as a device for the final product but for sure it is an alternative for a long-lasting method of dry mass content determination.

4 Conclusions

The tests aiming to check reliability of presented method results for processed cheese, have confirmed that the obtained results are valid. Conformity with the reference method, commonly used for this kind of determination, was achieved. The suggested method of dry mass content determination for processed cheese, carried out using a moisture analyzer, allows to shorten the drying time to about 9–16 min. The only difficulties regarding the suggested method are drying temperature optimization and analyzed sample size. Both these are crucial in order to provide a reliable result. The simulated cost analysis concerning one determination performed using both reference and alternative method, let the moisture analyzer to be qualified as an inexpensive tool. Reliability of the obtained results makes the moisture analyzer an attractive device that can be freely used in control of the final product, and also during the inter operational control. According to the authors, further tests would allow to obtain even lower deviation of the result when compared to the reference method values (improved repeatability).

Compliance with ethical standards

Human and animal rights This article does not contain any studies with human or animal subjects performed by any of the authors.

Conflict of interest Author Sławomir Janas is a member of "Radwag company, Radom - Poland" which supported the research.

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Table 5 Comparison for the drying methods costs for the processed cheese

Factors / Parameters	Unit	Reference method	Moisture analyzer method
Device price	€	4800	840
Price of accessories used for determination of moisture content	€	120	none
Power consumption	W	1600	450
Approximate operation time	h	4	0,5
Workforce cost	–	low	low
Risk and potential repair cost	–	low	low
Required workspace size	m^2	4	1

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