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A novel technique to control ice cream freezing by electrical characteristics analysis

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Abstract

The freezing process is very important in ice cream production affecting quality, taste and yield of the finished product. Batch freezer machines use different control techniques to tightly control the freezing process, based on monitoring of the temperature and/or the viscosity (i.e. consistency) of the product. Temperature control, however, features low sensitivity and need calibration for different product compositions, while product viscosity is essentially inferred from dasher motor load, sensitive to power line fluctuations, volume of product and dasher motor characteristics. In this context, this paper presents a novel technique based on measurements of the product electrical characteristics, tightly linked to temperature and viscosity. The experimental results presented in this work clearly indicate that the proposed technique provides a suitable, non destructive tool to monitor ice cream quality product that overcomes the drawbacks of the standard methods, thus representing an advance in the state of the art for freezing control.

21 Keywords: ice cream, freezing, dasher motor, impedance measurements.

1. Introduction

- 24 Ice cream has been appreciated in the western world since the 13th century, when Marco Polo
- 25 returned from the Far East Asia with water ice recipes. Over time, these water ices have evolved
- into today's popular frozen desserts (Marshall et al., 2003)(Quinzio, 2009).

- Nowadays the international ice-cream market has significant dimensions and faces prospects of
- continuous further growth worldwide (www.dairymark.com, 2008).
- Within ice cream production process, freezing is a very important operation strongly affecting the
- 30 quality, taste, and yield of the product (Marshall et al., 2003). While being frozen, the ice cream
- 31 mix is agitated in order to incorporate air and to limit the size of ice crystals that are formed, as
- 32 required to give smoothness in body and texture and satisfactory overrun in the ice cream (Marshall
- 33 et al., 2003) (Caillet et al., 2003) (Chang and Hartel, 2002) (Goff et al., 1999).
- 34 Typically, ice cream is produced fresh using a batch freezer and is then immediately extruded. The
- 35 finished product is then held and served at a temperature suitable for a highly viscous semi-frozen
- 36 state (Marshall et al., 2003).
- 37 Conventional batch freezer consists of a scraped-surface tubular heat exchanger, jacketed with
- 38 boiling refrigerants (such as hydrofluorocarbons) and containing a rotating dasher scraping the ice
- 39 off the inner surface and whipping the mix to incorporate air.
- 40 The mixture is frozen at a variable temperature (generally within the range -11 < T (°C) < -5),
- 41 according to type of process and composition, and very rapidly in order to obtain an even
- 42 distribution of the solid and aerated phases throughout the entire structure of the product, avoiding
- 43 separation of the solutes.
- 44 Batch freezers use different control techniques to ensure a tight control of the process, generally
- based on monitoring of the temperature and/or the viscosity (consistency) of the product, this latter
- being normally inferred from the current drawn by the dasher motor (I_{motor}).
- 47 Since the product temperature decreases very slowly during the final phase of the freezing process,
- 48 temperature monitoring, used in the past since the advent of viscosity control, normally provides
- 49 low sensitivity, leading to high dispersion in product softness. Moreover, the temperature at the end
- of the process strongly depends on ice cream composition and recipe as well as the actual
- 51 performance of the temperature sensor, that is placed at the bottom of the cylinder and can have bad
- 52 contact with the product.

Product viscosity control, normally carried out by monitoring I_{motor}, solves many of these problems. During the freezing process the product viscosity increases, and so does I_{motor}. Low values for this current indicates that the product is too soft or warm, while values higher than suitable threshold indicate that the product is too stiff or cold. This type of control too, however, has significant drawbacks, since measurements of I_{motor} are influenced by power line fluctuations, quantity of mixture in the freezing cylinder and dasher motor characteristics (thus, in practice, different stop process thresholds must be set for different type of electric motors). In this context, then, a reliable technique directly monitoring the product characteristics would be highly desirable and good candidates for the task are techniques based on measurements of the icecream electrical characteristics, known to be interesting indicators of product status, although only at macroscopic level, and to be function of product temperature and viscosity (Georges and Chen, 1986)(Hori et al., 1982). Moreover, these measurements can be performed with electronic circuits with stable power supply not influenced by motor type and power line fluctuation. Recently, electrochemical impedance spectroscopy has been used for non destructive monitoring in different areas of food processing and analysis, such as, for instance: determination of water and lipid content in meat (Chanet et al., 1999); determination of pH acidity and hardness in yogurt (Kitamuna et al., 2000); detection of mastitis in raw milk samples (Norberg et al., 2004) (Ferrero et al., 2002); measurement of microbial concentration in milk (Felice et al., 1999) (Piton and Dasen, 1988) (Piton and Rongvaux-Gaida, 1990), yogurt (Pirovano et al., 1995) and ice cream (Grossi et al., 2008) (Grossi et al., 2009) (Grossi et al., 2010); detection of protein concentration in label free immunosensors (Lin et al., 2010). Electrochemical Impedance Spectroscopy (EIS) essentially works as follows (Barsoukov and MacDonald, 2005): the analyzed sample is stimulated with a sinusoidal test signal of fixed amplitude (usually ranging from 10mV to few hundreds mV) and the sample impedance Z is measured in a definite range of frequencies. The acquired spectra can be represented using various graph types including Bode plots, where |Z| and Arg(Z) are plotted as function of frequency, or

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Nyquist plots where the imaginary component of Z is plotted versus the real one for different values of frequency. Data from different samples characterized by different parameters are compared to

test the correlation between product properties and electrical parameters.

82 In this paper EIS has been used to demonstrate that electrical parameters can provide information

about the ice cream status during freezing in a conventional batch freezer. To this purpose, data

acquired from EIS measurements have been compared to the freezer process parameters. In

particular, the correlation with the current drawn by the dasher motor has been studied.

In section 2 the measurement set up as well as the tested products are presented. Results are

discussed in section 3 while conclusions are drawn in section 4.

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2. Materials and methods

90 A conventional batch freezer (Fig. 1a) has been used with standard vanilla flavored ice cream mix

to test the correlation between the measured electrical parameters and the current drawn by the

dasher motor. The product temperature has been also monitored during the freezing process with a

PT100 thermistor placed within the cylinder in direct contact with the product.

The freezer (Coldelite Compacta Top 3002 RTX, Carpigiani Group) essentially represents a scraped

surface heat exchanger. The dasher (d=264 mm, L=255 mm) with the attached scraper blades

rotates within a cylindrical barrel, cooled by an evaporating agent (i.e. R-404A). Air is incorporated

and dispersed in the mix by the dasher. The mix is frozen and simultaneously scraped from the

cylindrical freezer wall by the scraper blades. The rotational speed of the dasher is 120 rpm.

When not differently specified, the experiments have been performed pre-cleaning the barrel with

hot water and completely filling the freezing chamber (with 7 liters of ice-cream mix).

In general, in batch freezers the process ends when I_{motor} reaches a threshold value I_{motor,TH}

dependent on the type of motor: for the freezer used in the work the default factory $I_{motor,TH}$ value is

6.5 A. The user can also vary the values of $I_{motor,TH}$ suggested by machine producer (within about \pm

10%) to achieve the desired product softness with specific mix compositions: higher values of $I_{motor,TH}$ are used for products featuring high concentrations for sugar and/or total solids.

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2.1 Experimental set up

The front door of the batch freezer (Fig. 1 b) has been modified so as to use two adjacent stainless steel cylinders as a couple of electrodes for the electrochemical measures. Such electrodes have been connected to the input port of the impedance analyzer Agilent E4980A, capable of measuring the electrical characteristics of the sample on a range of frequencies from 20 Hz to 2 MHz. A PC laptop acquires the measured data from the impedance analyzer via a USB interface and from the batch freezer via a serial RS232 port. All programs for data acquisition, graphing and data filing have been realized with National Instruments LabVIEW software. A schematic representation of the experimental set up is shown in Fig. 1 (c). Preliminary measurements have been carried out to investigate the optimal experimental parameters, namely values for amplitude and frequencies of the input test voltage. In general, in EIS measurements the amplitude of the input voltage (Vin) is kept at the lowest value compatible with the need of measurement reliability, as lower voltages produce lower perturbations to the sample under test and quasi linear responses (Darowicki, 1995). In our case, preliminary measurements suggested to use an amplitude of 100 mV for the test signal: lower values resulted inadequate signal-noise ratios (due to the strong electromagnetic noise produced by the motor), while higher values resulted in stronger sample alterations with no improvements in measurement reliability. As for frequency of the test signal, measurements have been carried out with values logarithmically spaced in the range 20 Hz - 10 KHz. Fig. 2 (a) and (b) show plots of |Z| and Arg(Z) vs. time for different frequencies. Since, however, the different assays are strongly influenced by the dynamics of the refrigerating system (see Supplementary Material for more details), the time origin for each assay has been set at the beginning of the product temperature decrease. As can be seen the |Z| curve is almost independent of the frequency for the test signal. On the contrary, Arg(Z) exhibits high sensitivity to such a parameter: in particular, the signal to noise ratio decreases with frequency and for values higher than 100 Hz measurements become substantially unreliable. Moreover, as reported in the Supplementary Material, Nyquist plots indicate that higher frequencies lead to high noise levels and poor measurement reproducibility. Thus, in our experiments test signal frequency has been set at 20 Hz.

The current drawn by the dasher motor and the product temperature during freezing are represented

The current drawn by the dasher motor and the product temperature during freezing are represented as function of time in Fig. 2 (c) and Fig. 2 (d), respectively. As can be seen, product temperature decreases at the beginning of the freezing process and saturates at a value ranging from -9°C to -5°C, clearly indicating that temperature measurement can only provide approximate results and is unsuitable for accurate process control.

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- 142 2.2 Ice cream mix
- 143 A standard vanilla ice cream mix (Mondi nel Mondo, Rome, Italy, www.mondinelmondo.it) has
- been used for the experiments. The ingredients of the mix are sugar, coconut oil, dextrose, glucose
- powder, milk proteins, stabilizers (E412, E410, E466), emulsifiers (E471, E473), flavoring in the
- 146 composition of 17.5 Kg by powder and 30 liters by water. The ice cream recipe is as follows: fat
- 147 content 11%, milk solids-not-fat 11%, sugar 10%, corn syrup solids 5%, stabilizers/emulsifiers
- 148 0.5%, total solids 37.5%.
- The ice cream mix production has been carried out using a Carpigiani Pastomaster RTL machine to
- prepare, pasteurize and age ice cream mixes. The basic steps in the manufacturing are as follows:
- mixing of ingredients (i.e. mixing powder with water in the tank of the pasteurizer);
- pasteurization at 65 °C for 30 minute (automatically managed by the pasteurizer);
- cooling to 4 °C (automatically managed by the pasteurizer);
- ageing at 4 °C for 10 hours (automatically managed by the pasteurizer).

155 Small batches of mix were taken from the pasteurizer and put into the batch freezer; then, the dasher 156 was started and the refrigerant system turned on. Finally, at the end of the freezing process, the ice cream was extracted from the freezer and the 157 158 machine was cleaned. 159 160 2.3 Statistical analysis 161 The obtained data have been analyzed using Microsoft EXCEL statistical tools. Measured values of 162 |Z|, Arg(Z) and $\Delta Arg(Z)$ (difference between two consecutive measurements of Arg(Z)) have been represented as function of the corresponding values of I_{motor} and correlation between the variables 163 164 has been studied by means of linear regression analysis (Mason et al., 2003). Multiple regression 165 analysis has been carried out with Microsoft EXCEL add-on Prentice Hall PhSTAT v 1.4. 166 167 3. Results and discussion The correlation between electrical parameters and I_{motor} in the experiments described in the previous 168 Section has been intensively investigated. 169 170 Fig. 3 (a) shows the experimental data |Z| vs. I_{motor} clearly exhibiting a linear relationship between the two variables. The linear regression line equation as well as the determination factor R² have 171 172 been calculated. The dispersion among the predicted value is not uniform on the entire range of 173 I_{motor} , in particular it increases when $I_{motor} > 5$ A. 174 Fig. 3 (b) shows the experimental data Arg(Z) vs. I_{motor}. At the beginning of the freezing process, 175 when temperature is not very low and mix is still in the liquid state, Arg(Z) increases rapidly. Later, 176 the mix density increases, producing first a slower increase and later a saturation of Arg(Z). For 177 $I_{motor} > 4$ A (when freezing has already started) the relation Arg(Z) vs. I_{motor} is essentially linear, as

clearly indicated in Fig. 3 (c) representing data obtained for I_{motor} values in the range 3.5 - 7.5 A.

The linear regression line equation for the measured data as well as the determination factor R² are

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- also presented. From Fig. 3 (b) it is also evident that dispersion on the estimated value of Arg(Z) is
- lower at the end of the freezing process.
- The values of $\Delta Arg(Z)$ increase rapidly at the beginning of the freezing process (low I_{motor} values),
- reach a maximum at $I_{motor} \approx 2.5$ A, then decreases as freezing continues. In Fig. 3 (d) values of
- $\Delta Arg(Z)$ are plotted against I_{motor} in the range 3 7.5 A. The linear regression line equation as well
- as the determination factor R^2 are presented too. As can be seen, data dispersion is initially higher
- and decreases as the freezing process continues.
- As for the correlation between product temperature and I_{motor}, the results in the Supplementary
- 188 Material show that it is very poor.
- From the scatter plots of Fig. 3 the threshold value for the electrical parameters to stop the freezing
- 190 process ($|Z|_{TH}$, $Arg(Z)_{TH}$ and $\Delta Arg(Z)_{TH}$) can be estimated from the regression line equation as
- 191 function of the threshold I_{motor,TH}. Since, for the batch freezer used in the experiments, the producer
- suggested $I_{motor,TH} = 6.5A$, the corresponding thresholds for the electrical parameters are $|Z|_{TH} =$
- 193 755.3 Ω , Arg(Z)_{TH} = -13.58° and Δ Arg(Z)_{TH} = 2.26°.
- The repeatability of the experiments has been tested by means of 8 freezing assays performed under
- the same conditions and the results, as well as statistics for the time to reach the stop process
- threshold, are shown in Fig. 4. As can be seen, the freezing control using |Z| leads to more
- repeatable curves and minimum dispersion on the stopping time (Δ time = 1.85 minutes), than those
- 198 exploiting measurements of Arg(Z) ($\Delta time = 3.6 \text{ minutes}$) and $\Delta Arg(Z)$ ($\Delta time = 3.1 \text{ minutes}$).
- These latter methods, however, exhibit smaller dispersion than straightforward I_{motor} control (Δ time
- = 3.88 minutes).
- 201 Multivariate regression analysis has been carried out using best-subset procedure to investigate if
- 202 expressing I_{motor} as linear function of more than a single electrical parameter significantly increases
- 203 data correlation. To this purpose, it is found that using both |Z| and Arg(Z) significantly improves
- the correlation (R^2 =0.79 instead of R^2 =0.69), while adding $\Delta Arg(Z)$ produces only a negligible
- increase. Thus, in the following, only measurements of |Z| and Arg(Z) are considered.

The experiments described above were performed by cleaning the freezer with hot water before the start of the process (so as to eliminate residuals of ice cream from previous assays and making the initial temperature as homogeneous as possible) and filling the barrel always with the same quantity of mix (corresponding to the maximum capacity of 7 liters). Since this is not the case during the normal operation of a batch freezer, the effects of consecutive freezing processes without cleaning as well as loading the freezer with different mix quantities have been studied. To this purpose, three freezing procedures have been carried out in a row without cleaning the machine barrel between consecutive assay and the results are presented in Fig. 5. As can be seen, values of |Z| are not significantly affected by the absence of barrel cleaning between different freezing processes. On the contrary, Arg(Z) exhibits significantly higher values only at the beginning of the freezing process when the machine barrel is not cleaned from the previous operation (possibly because of the presence of residuals of frozen ice cream between electrodes). As freezing proceeds, however, these differences disappear and the data are no longer dependent on repeated machine cleaning. Therefore, lack of cleaning between different freezings in practice does not affect the reliability of electrical parameter measurements to determine the end of the freezing process. A typical situation where the freezing process fails to control the product stiffness is when the ice cream freezes on the blades of the dasher motor, a problem more likely to occur when the cylinder is fully loaded and the temperature is very low at the beginning of the process. To investigate the capability of the type of control proposed in this work to overcome this problem, |Z|, Arg(Z) and I_{motor} have been monitored during freezing assays featuring ice cream freezing on the blades of the dasher motor. The curves shown in Fig. 6 (a), (b) and (c) indicate that the values of I_{motor} remain always lower than the threshold of 6.5 A, while |Z| and Arg(Z) reach their stop process thresholds essentially at the same times than in the "standard" freezing experiments of Fig. 4. Therefore, the combined monitor of both electrical parameters and I_{motor} can be used to detect the occurrence of the

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231 problem of ice-cream freezing on the scraping blades (see Supplementary Material for |Z| and 232 Arg(Z) plotted vs. I_{motor}), allowing the user to promptly take countermeasures. 233 Fig. 7 (a), (b) and (c) show the time evolution of |Z|, Arg(Z) and I_{motor} in the case of the freezer 234 filled with four different volumes of ice cream mix: 3 liters (minimum value of freezing capacity), 4 liters, 5 liters and 7 liters (maximum value of freezing capacity). Both |Z| and I_{motor} do not reach the 235 236 corresponding stop process thresholds for the 3 liters case, while Arg(Z) is almost not influenced by 237 mix volume and provides good control also at lowest load. As can be seen, the |Z| vs. time curve 238 depends on the mix volume. In particular, in the case of 3 liters, |Z| initially increases (for the first 4 minutes), then slightly 239 240 decreases (from 4 to 6 minutes) while later increases again. This irregular behavior could be 241 explained as follows. When the ice-cream volume is low, electrodes are only partially covered by 242 the ice-cream mix, thus resulting in less conductivity and higher |Z|. When freezing begins, the 243 product temperature lowers producing an increase in both mix volume and electrodes coverage: |Z| 244 decreases. When, instead, ice-cream volume increases no more |Z| is bound to increase with 245 decreasing temperature. Thus the irregular behavior of |Z| can be the results of the opposite events 246 of increasing electrodes coverage and product freezing (decreasing and increasing |Z|, respectively). Even if measurements of neither |Z| nor I_{motor} provide reliable control for the freezing process in the 247 248 case of the minimum volume (3 liters), the former type of monitor is preferable. In fact, on the 249 contrary of motor stress and I_{motor} that depend on product volume, the problem of |Z| is related to 250 electrode area coverage and can be solved by suitable placing of the electrodes so as they are 251 entirely covered by product even in the case of minimum mix volume. 252 The reason because |Z| is sensitive to electrode coverage while Arg(Z) is not, can be understood 253 considering the electrical circuit for a couple of electrodes of length L partially covered by a 254 conductive media, up to a length l < L (Fig. 7 (d)).

Denoting R_{AIR} and R_{MIX} the resistances of the air and the media, respectively, since $R_{AIR} >> R_{MIX}$

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the system impedance can be described as:

$$257 Z = R_{MIX} + \frac{1}{i\omega C_{MIX}} , (1)$$

258 thus:

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$$|Z| = \sqrt{R^2_{MIX} + \left(\frac{1}{\omega C_{MIX}}\right)^2}$$
, (2)

$$260 Arg(Z) = -Arctg\left(\frac{1}{\omega R_{MIX} C_{MIX}}\right) . (3)$$

- Denoting as R^*_{MIX} and C^*_{MIX} the resistance and capacitance for the electrodes covered with unit
- length of conductive media, R*_{MIX} and C*_{MIX} are independent on the electrodes coverage level and
- 263 thus on the mix volume. It is $R_{MIX} = \frac{R^*_{MIX}}{l}$ and $C_{MIX} = l \cdot C^*_{MIX}$, hence:

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$$|Z| = \frac{1}{l} \cdot \sqrt{R^*_{MX} + \left(\frac{1}{\omega C^*_{MX}}\right)^2}$$
, (4)

$$265 Arg(Z) = -Arctg\left(\frac{1}{\omega R^*_{MX} C^*_{MX}}\right) . (5)$$

- Eq. 4 and 5 indicate that |Z| decreases as 1 increase, while Arg(Z) is independent of 1.
- To support this interpretation, |Z| and Arg(Z) have been measured in the frequency range 20 Hz 10
- 268 KHz in experiments featuring the machine filled with different volumes of ice cream mix and with
- the dasher motor turned off to guarantee a constant level of electrode coverage. The results
- 270 (presented in the Supplementary Material), fully confirm the electrical model of Fig 7 (d).
- 271 Preliminary measurements have also been carried out on other three ice cream mixes, different for
- composition and producers, and the results (presented in the Supplementary Material) are in good
- agreement with those described above in this paper, clearly indicating that the technique proposed
- 274 in this paper can be successfully applied to different mixes without the need of individual
- 275 calibration.

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277 **5. Conclusions**

In this paper a novel technique to control the freezing process of ice creams in conventional batch freezer has been presented. The new method, based on the measurement of the product electrical parameters, |Z| and Arg(Z), by a couple of stainless steel electrodes (for instance placed on the front door of the machine), presents significant advantages compared with the current technique based on monitoring of the current drawn by the dasher motor. This, in essence, because the electrical parameters are an indicators of product status, although only at macroscopic level, hence, contrary to the current drawn by the dasher motor, are insensitive to power line fluctuations and electric motor characteristics, as well as independent of the volume of ice-cream within the machine (provided suitable placing of the electrodes so as they are completely covered with product even in the case of minimum mix volume).

The experimental data presented in the paper show a good linear correlation of both |Z| and Arg(Z) with the current drawn by the dasher motor, with |Z| being the best choice as parameter to be

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represents an economical and reliable technique advancing the state of the art in the field.

On the whole, the control of ice cream freezing process by electrical parameters monitoring

monitored because of higher data correlation and repeatability.

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(a)

(b)

LCZ meter Agilent E4980A

USB port

| Z| 250,00 \(\Omega \)
| Arg(Z) -45,00 Deg |
| electrodes input to LCZ meter |
| Serial port |
| COLDELITE - COMPACTA TOP - 3002 RTX

Fig. 1 Industrial batch freezer used in the experiments (a), frontal grid of the machine (b) and a scheme representing the set-up used in the experimental.

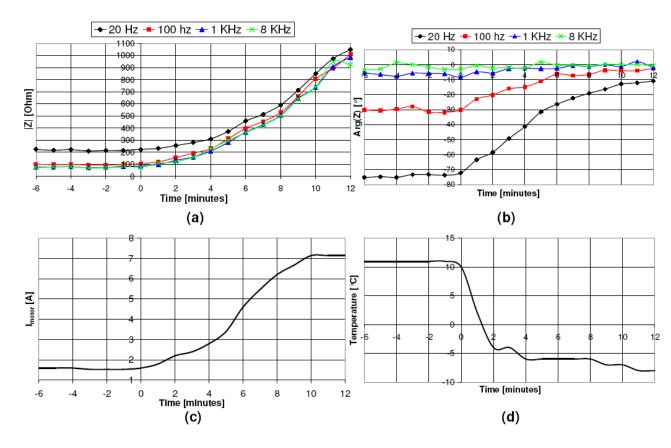


Fig. 2 Curves of |Z| and Arg(Z) vs. time measured for different frequencies of the test signal (Fig. a and b, respectively). Current drawn by the dasher motor (c) and product temperature (d) as function of time during the freezing process for a vanilla flavored ice cream mix.

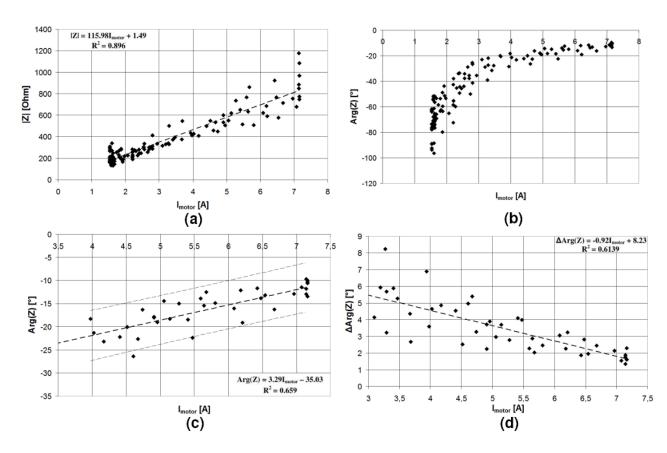


Fig. 3 Curves of |Z| (a), Arg(Z) and $\Delta Arg(Z)$ (d) vs. I_{motor} . Two curves Arg(Z) vs. I_{motor} are presented for the entire range values of I_{motor} (b) and for values in the range 3.5A to 7.5A (c).

1200 time_{mesn} = 9.79 min $\sigma_{\rm time}$ = 0.82 min Δ time = 1.85 min 1000 [Ohm] Arg(Z) [*] |Z|_{TH} 600 ∆time -10 10 15 -5 Time [minutes] Time [minutes] (a) (b) ... = 9.66 min Imotor [A] ∆time ∆time -5 10 15 10 -10 Time [minutes] Time [minutes]

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Fig. 4 Repetition trials for the ice cream freezing process. The values of |Z|, Arg(Z), $\Delta Arg(Z)$ and I_{motor} are plotted vs. time, while statistics for the stop process time are presented. The legend for the trial number is as follows: 1^{st} (\blacklozenge , dark blue), 2^{nd} (\blacksquare , pink), 3^{rd} (\blacktriangle , yellow), 4^{th} (\bigstar , light blue), 5^{th} (\bigstar , purple), 6^{th} (\blacklozenge , brown), 7^{th} (I, dark green), 8^{th} (\lnot , blue).

(c)

(d)

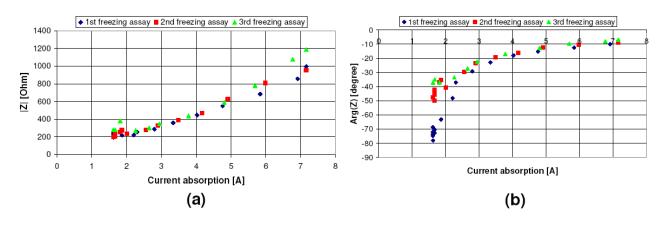


Fig. 5 |Z| and Arg(Z) vs. I_{motor} for freezing processes carried out without cleaning the machine barrel between consecutive assays.

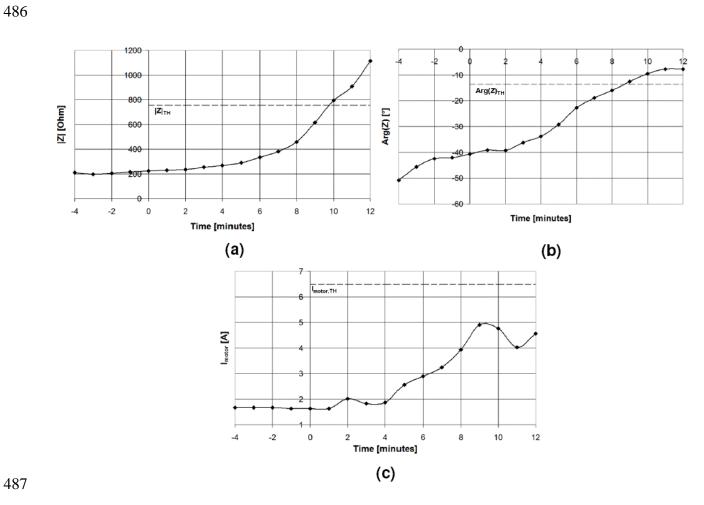


Fig. 6 Monitored values of |Z| (a), Arg(Z) (b) and I_{motor} (c) for a freezing process when the mix freezes on the blades of the dasher motor.

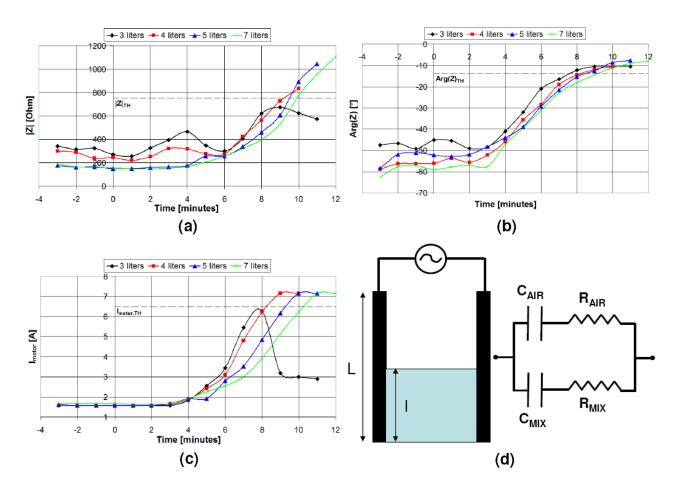


Fig. 7 Curves of |Z|, Arg(Z) and I_{motor} vs. time for freezing processes carried out filling the machine barrel with different volumes of ice cream mix (Fig. a, b and c, respectively). Electrical model for the system formed by a couple of electrodes only partially covered by conductive media (d).

Supplementary Material

A novel technique to control ice cream freezing by electrical

characteristics analysis

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The Nyquist plot of Fig. S1 can be also used to validate the electrical model for the system product-

electrodes. A model composed of the series of a resistance R_{MIX} (accounting for the product

conductivity) and a capacitance C_{MIX} (essentially due to the capacitive product-electrode interface)

This is consistent with Fig. S1 where Re(Z) is essentially frequency independent and Im(Z)

 $\operatorname{Im}(Z) = -\frac{1}{\omega C_{MX}}$

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at high frequency is significantly higher than at the beginning of the process.

 $Re(Z) = R_{MIX}$

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EIS monitoring of the freezing process. The ice cream mix has been subjected to freezing process

and the electrical parameters |Z| and Arg(Z) have been measured for the frequency range 20Hz -10KHz at time intervals of 1 minute. In Fig. S1 the Nyquist plot for EIS measurements are shown for different acquisition times. As can be seen, higher frequency measurements result in lower signal-to-noise ratio, poor reproducibility and, in general, higher dispersion than the corresponding low frequency measurements (due to the electromagnetic noise generated by the dasher motor). This, in particular, affects the last phase of the freezing process when the measurements dispersion

has been used. Thus:

 $Z = R_{MIX} + \frac{1}{j\omega C_{MIX}}$

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decreases as the frequency increases.

-Im(Z) [Ohm]

Fig. S1 Nyquist plots for different acquisition times (in minutes) during the freezing process.

Re(Z) [Ohm]

Repetition of the freezing assays. Different freezing assays has been carried out using the same ice-cream mix (described in the Section "Materials and Methods") and conditions (cylinder washed with hot water between assays and filled with 7 liters of mix). Nevertheless, the curves of both the electrical parameters and current drawn by the dasher motor vs acquisition time presented poor repeatability. This must be ascribed to the dynamics of the freezing system. As can be seen in Fig. S2, where the temperature of the product is plotted as function of the acquisition time, the time t* when the product temperature begins to decrease presents an high dispersion (with values of t* ranging from 2.7 minutes to 8.8 minutes). Thus, to eliminate the dispersion due to the freezing system dynamics, the time origin has been set to the instant the product temperature begins to decrease.

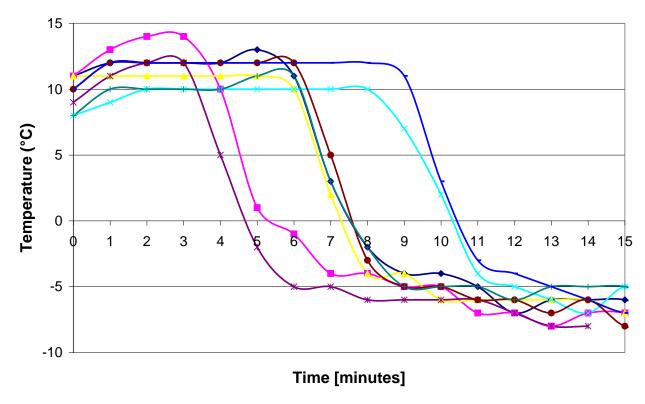
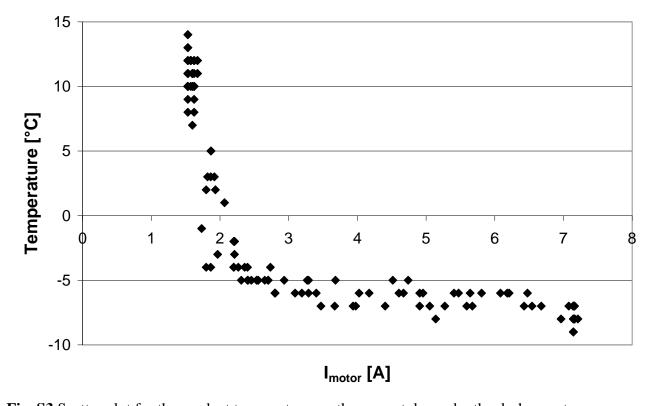


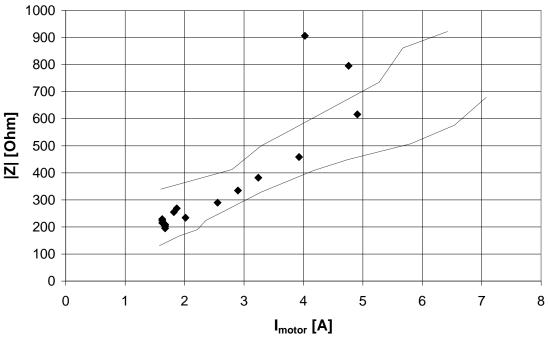
Fig. S2 Product temperature vs. acquisition time for different freezing assays.

<u>Correlation between product temperature and current drawn by the dasher motor</u>. The product temperature is monitored during the freezing assay by the sensor temperature PT100, placed in direct contact with the product. In Fig. S3 the product temperature is plotted versus the current drawn by the dasher motor I_{motor} . As can be seen, very poor correlation exists between the two parameters. The product temperature decrease at the beginning of the freezing process and then saturates at a value ranging from -9°C to -5°C.



 $\textbf{Fig. S3} \ \ \textbf{Scatter plot for the product temperature vs. the current drawn by the dasher motor.}$

Detection of ice cream mix freezing on the blades of the dasher motor by combined monitoring of electrical characteristics and current drawn by the dasher motor. This problem can happen during the freezing assay in particular when the freezer cylinder is loaded with maximum mix volume (7 liters) and the chamber temperature is very low already at the beginning of the process. This event must be promptly detected since under this condition the proper control of product stiffness is compromised. In Fig. S4 the monitored values for |Z| and Arg(Z) are plotted versus the corresponding I_{motor} values for a freezing assay featuring this particular problem. Lower and higher bounds from repeatability tests are also shown. As can be seen, as the mix freezes on the blades of the dasher motor, values of I_{motor} decreases while |Z| and Arg(Z) increases. This causes the measured data to fall off the safe working area represented by dashed lines in Fig. S4. Thus, combined monitoring of |Z|, Arg(Z) and I_{motor} can promptly detect this problem.



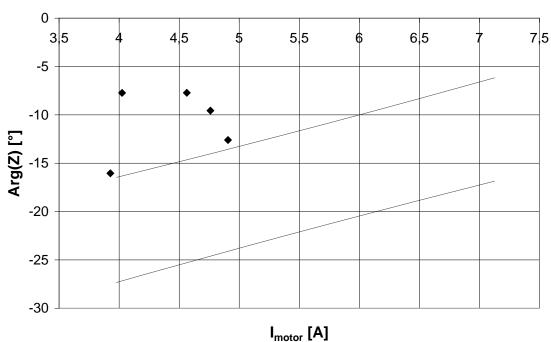
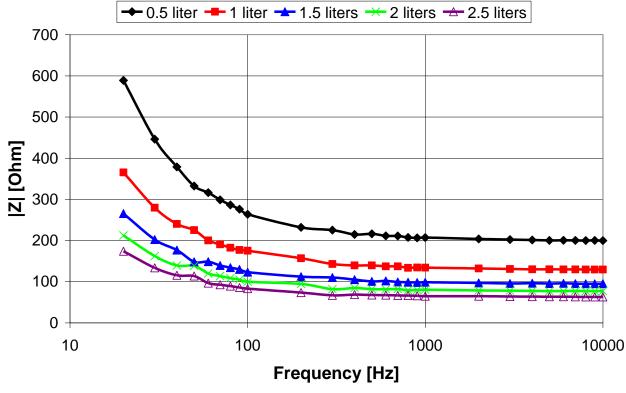


Fig. S4 Plots of |Z| and Arg(Z) vs. I_{motor} for a freezing assay featuring mix freezing on the blades of the dasher motor.

<u>EIS analysis of ice cream mix with different levels of electrodes area coverage</u>. As a further proof of the dependence of electrical parameters on the electrode area coverage, the machine has been loaded with different volumes of ice cream mix (with the dasher motor turned off to guarantee a constant level of electrode coverage) and |Z|, Arg(Z) measured on the frequency range 20 Hz - 10 KHz. The results, presented in Fig. S5, show how while |Z| is a strong function of electrodes area coverage, Arg(Z) is essentially independent.



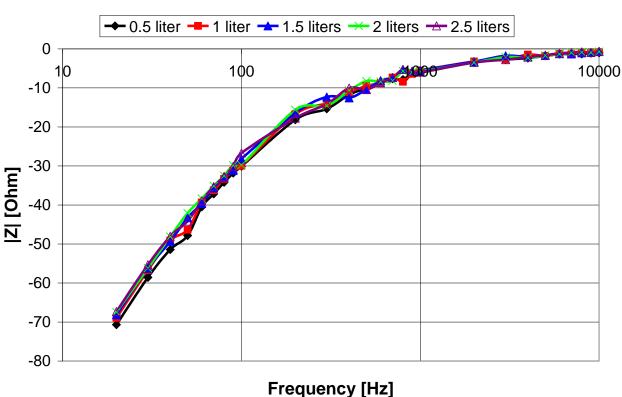


Fig. S5 |Z| and Arg(Z) as function of frequency for different ice cream mix volumes.

 <u>Measurements on different ice cream mixes</u>. Preliminary measurements have been carried out on a limited number of ice cream mix of different compositions and producers to test the correlation of the electrical parameters |Z| and Arg(Z) with the current drawn by the dasher motor and compare the results with those obtained with the test mix described in the Materials and Methods section.

The recipes for the three product tested, hereafter named Ice Cream Mix A, B and C, are as follows.

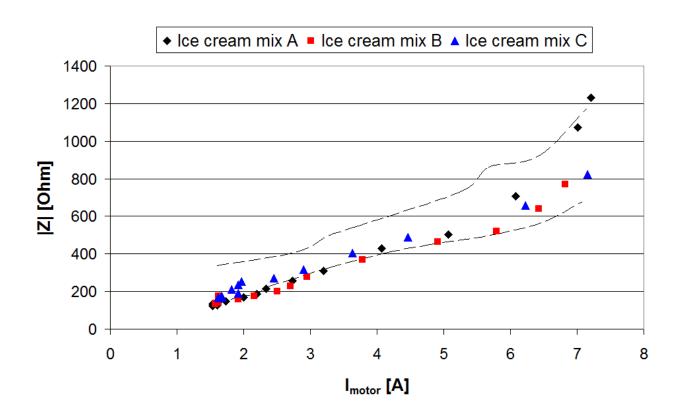
Ice Cream Mix A: fat content 6%, milk solids-not-fat 12.5%, sugar 12%, corn syrup solids 4%, stabilizers/emulsifiers 0.4%, total solids 34.9%.

Ice Cream Mix B: fat content 10%, milk solids-not-fat 11%, sugar 12%, corn syrup solids 3%, stabilizers/emulsifiers 0.4%, total solids 36.4%.

Ice Cream Mix C: fat content 6%, milk solids-not-fat 11%, sugar 11%, corn syrup solids 5%, stabilizers/emulsifiers 0.5%, total solids 33.5%.

Ice Cream Mix A is produced by Berglandmilch (Austria, www.berglandmilch.at), while Ice Cream Mix B and C by Mondi nel Mondo (Rome, Italy, www.mondinelmondo.it).

In Fig. S6 the electrical parameters |Z| and Arg(Z) are plotted vs current drawn by the dasher motor. Dashed lines represent the lower and higher bounds for the test mix described in the "Materials and Methods" section resulting from the scatter plots of Fig. 3 in the manuscript. As can be seen, measurements present good agreements with data obtained with the test mix, with only slightly lower values of |Z| at the beginning of the freezing process.



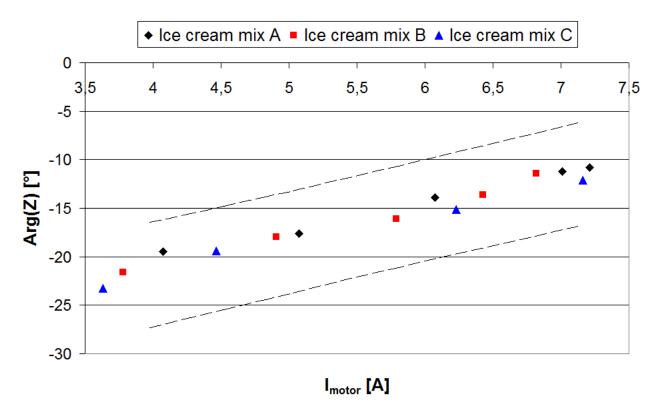


Fig. S6 Scatter plots for |Z| and Arg(Z) as function of the current drawn by the dasher motor for different ice cream mixes.