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► To cite this version:

Marco Grossi, Roberto Lazzarini, Massimo Lanzoni, Bruno Riccò. A novel technique to control ice cream freezing by electrical characteristics analysis. *Journal of Food Engineering*, 2011, 106 (4), pp.347-354. 10.1016/j.jfoodeng.2011.05.035 . hal-01276543

HAL Id: hal-01276543

<https://hal.science/hal-01276543>

Submitted on 25 Feb 2016

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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.

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

1. Introduction

Ice cream has been appreciated in the western world since the 13th century, when Marco Polo 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).

27 Nowadays the international ice-cream market has significant dimensions and faces prospects of
28 continuous further growth worldwide (www.dairymark.com, 2008).

29 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 $-11 < T (^{\circ}\text{C}) < -5$),
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
45 based on monitoring of the temperature and/or the viscosity (consistency) of the product, this latter
46 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
50 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.

53 Product viscosity control, normally carried out by monitoring I_{motor} , solves many of these problems.
54 During the freezing process the product viscosity increases, and so does I_{motor} . Low values for this
55 current indicates that the product is too soft or warm, while values higher than suitable threshold
56 indicate that the product is too stiff or cold. This type of control too, however, has significant
57 drawbacks, since measurements of I_{motor} are influenced by power line fluctuations, quantity of
58 mixture in the freezing cylinder and dasher motor characteristics (thus, in practice, different stop
59 process thresholds must be set for different type of electric motors).

60 In this context, then, a reliable technique directly monitoring the product characteristics would be
61 highly desirable and good candidates for the task are techniques based on measurements of the ice-
62 cream electrical characteristics, known to be interesting indicators of product status, although only
63 at macroscopic level, and to be function of product temperature and viscosity (Georges and Chen,
64 1986)(Hori et al., 1982). Moreover, these measurements can be performed with electronic circuits
65 with stable power supply not influenced by motor type and power line fluctuation.

66 Recently, electrochemical impedance spectroscopy has been used for non destructive monitoring in
67 different areas of food processing and analysis, such as, for instance: determination of water and
68 lipid content in meat (Chanet et al., 1999); determination of pH acidity and hardness in yogurt
69 (Kitamuna et al., 2000); detection of mastitis in raw milk samples (Norberg et al., 2004) (Ferrero et
70 al., 2002); measurement of microbial concentration in milk (Felice et al., 1999) (Piton and Dasen,
71 1988) (Piton and Rongvaux-Gaida, 1990), yogurt (Pirovano et al., 1995) and ice cream (Grossi et
72 al., 2008) (Grossi et al., 2009) (Grossi et al., 2010); detection of protein concentration in label free
73 immunosensors (Lin et al., 2010).

74 Electrochemical Impedance Spectroscopy (EIS) essentially works as follows (Barsoukov and
75 MacDonald, 2005): the analyzed sample is stimulated with a sinusoidal test signal of fixed
76 amplitude (usually ranging from 10mV to few hundreds mV) and the sample impedance Z is
77 measured in a definite range of frequencies. The acquired spectra can be represented using various
78 graph types including Bode plots, where $|Z|$ and $\text{Arg}(Z)$ are plotted as function of frequency, or

79 Nyquist plots where the imaginary component of Z is plotted versus the real one for different values
80 of frequency. Data from different samples characterized by different parameters are compared to
81 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
83 about the ice cream status during freezing in a conventional batch freezer. To this purpose, data
84 acquired from EIS measurements have been compared to the freezer process parameters. In
85 particular, the correlation with the current drawn by the dasher motor has been studied.

86 In section 2 the measurement set up as well as the tested products are presented. Results are
87 discussed in section 3 while conclusions are drawn in section 4.

88

89 **2. Materials and methods**

90 A conventional batch freezer (Fig. 1a) has been used with standard vanilla flavored ice cream mix
91 to test the correlation between the measured electrical parameters and the current drawn by the
92 dasher motor. The product temperature has been also monitored during the freezing process with a
93 PT100 thermistor placed within the cylinder in direct contact with the product.

94 The freezer (Coldelite Compacta Top 3002 RTX, Carpigiani Group) essentially represents a scraped
95 surface heat exchanger. The dasher ($d=264$ mm, $L=255$ mm) with the attached scraper blades
96 rotates within a cylindrical barrel, cooled by an evaporating agent (i.e. R-404A). Air is incorporated
97 and dispersed in the mix by the dasher. The mix is frozen and simultaneously scraped from the
98 cylindrical freezer wall by the scraper blades. The rotational speed of the dasher is 120 rpm.

99 When not differently specified, the experiments have been performed pre-cleaning the barrel with
100 hot water and completely filling the freezing chamber (with 7 liters of ice-cream mix).

101 In general, in batch freezers the process ends when I_{motor} reaches a threshold value $I_{\text{motor,TH}}$
102 dependent on the type of motor: for the freezer used in the work the default factory $I_{\text{motor,TH}}$ value is
103 6.5 A. The user can also vary the values of $I_{\text{motor,TH}}$ suggested by machine producer (within about \pm

104 10%) to achieve the desired product softness with specific mix compositions: higher values of
105 $I_{\text{motor,TH}}$ are used for products featuring high concentrations for sugar and/or total solids.

106

107 *2.1 Experimental set up*

108 The front door of the batch freezer (Fig. 1 b) has been modified so as to use two adjacent stainless
109 steel cylinders as a couple of electrodes for the electrochemical measures. Such electrodes have
110 been connected to the input port of the impedance analyzer Agilent E4980A, capable of measuring
111 the electrical characteristics of the sample on a range of frequencies from 20 Hz to 2 MHz. A PC
112 laptop acquires the measured data from the impedance analyzer via a USB interface and from the
113 batch freezer via a serial RS232 port. All programs for data acquisition, graphing and data filing
114 have been realized with National Instruments LabVIEW software. A schematic representation of
115 the experimental set up is shown in Fig. 1 (c).

116 Preliminary measurements have been carried out to investigate the optimal experimental
117 parameters, namely values for amplitude and frequencies of the input test voltage. In general, in EIS
118 measurements the amplitude of the input voltage (V_{in}) is kept at the lowest value compatible with
119 the need of measurement reliability, as lower voltages produce lower perturbations to the sample
120 under test and quasi linear responses (Darowicki, 1995). In our case, preliminary measurements
121 suggested to use an amplitude of 100 mV for the test signal: lower values resulted inadequate
122 signal-noise ratios (due to the strong electromagnetic noise produced by the motor), while higher
123 values resulted in stronger sample alterations with no improvements in measurement reliability.

124 As for frequency of the test signal, measurements have been carried out with values logarithmically
125 spaced in the range 20 Hz - 10 KHz.

126 Fig. 2 (a) and (b) show plots of $|Z|$ and $\text{Arg}(Z)$ vs. time for different frequencies. Since, however,
127 the different assays are strongly influenced by the dynamics of the refrigerating system (see
128 Supplementary Material for more details), the time origin for each assay has been set at the
129 beginning of the product temperature decrease. As can be seen the $|Z|$ curve is almost independent

130 of the frequency for the test signal. On the contrary, $\text{Arg}(Z)$ exhibits high sensitivity to such a
131 parameter: in particular, the signal to noise ratio decreases with frequency and for values higher
132 than 100 Hz measurements become substantially unreliable. Moreover, as reported in the
133 Supplementary Material, Nyquist plots indicate that higher frequencies lead to high noise levels and
134 poor measurement reproducibility. Thus, in our experiments test signal frequency has been set at
135 20 Hz.

136 The current drawn by the dasher motor and the product temperature during freezing are represented
137 as function of time in Fig. 2 (c) and Fig. 2 (d), respectively. As can be seen, product temperature
138 decreases at the beginning of the freezing process and saturates at a value ranging from -9°C to -
139 5°C , clearly indicating that temperature measurement can only provide approximate results and is
140 unsuitable for accurate process control.

141

142 *2.2 Ice cream mix*

143 A standard vanilla ice cream mix (Mondi nel Mondo, Rome, Italy, www.mondinelmondo.it) has
144 been used for the experiments. The ingredients of the mix are sugar, coconut oil, dextrose, glucose
145 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%.

149 The ice cream mix production has been carried out using a Carpigiani Pastomaster RTL machine to
150 prepare, pasteurize and age ice cream mixes. The basic steps in the manufacturing are as follows:

- 151 - mixing of ingredients (i.e. mixing powder with water in the tank of the pasteurizer);
- 152 - pasteurization at 65°C for 30 minute (automatically managed by the pasteurizer);
- 153 - cooling to 4°C (automatically managed by the pasteurizer);
- 154 - 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.

157 Finally, at the end of the freezing process, the ice cream was extracted from the freezer and the
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 $|Z|$, $\text{Arg}(Z)$ and $\Delta\text{Arg}(Z)$ (difference between two consecutive measurements of $\text{Arg}(Z)$) have been
163 represented as function of the corresponding values of I_{motor} and correlation between the variables
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**

168 The correlation between electrical parameters and I_{motor} in the experiments described in the previous
169 Section has been intensively investigated.

170 Fig. 3 (a) shows the experimental data $|Z|$ vs. I_{motor} clearly exhibiting a linear relationship between
171 the two variables. The linear regression line equation as well as the determination factor R^2 have
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 $I_{\text{motor}} > 5$ A.

174 Fig. 3 (b) shows the experimental data $\text{Arg}(Z)$ 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, $\text{Arg}(Z)$ increases rapidly. Later,
176 the mix density increases, producing first a slower increase and later a saturation of $\text{Arg}(Z)$. For
177 $I_{\text{motor}} > 4$ A (when freezing has already started) the relation $\text{Arg}(Z)$ vs. I_{motor} is essentially linear, as
178 clearly indicated in Fig. 3 (c) representing data obtained for I_{motor} values in the range 3.5 - 7.5 A.

179 The linear regression line equation for the measured data as well as the determination factor R^2 are

180 also presented. From Fig. 3 (b) it is also evident that dispersion on the estimated value of $\text{Arg}(Z)$ is
181 lower at the end of the freezing process.

182 The values of $\Delta\text{Arg}(Z)$ increase rapidly at the beginning of the freezing process (low I_{motor} values),
183 reach a maximum at $I_{\text{motor}} \approx 2.5$ A, then decreases as freezing continues. In Fig. 3 (d) values of
184 $\Delta\text{Arg}(Z)$ are plotted against I_{motor} in the range 3 - 7.5 A. The linear regression line equation as well
185 as the determination factor R^2 are presented too. As can be seen, data dispersion is initially higher
186 and decreases as the freezing process continues.

187 As for the correlation between product temperature and I_{motor} , the results in the Supplementary
188 Material show that it is very poor.

189 From the scatter plots of Fig. 3 the threshold value for the electrical parameters to stop the freezing
190 process ($|Z|_{\text{TH}}$, $\text{Arg}(Z)_{\text{TH}}$ and $\Delta\text{Arg}(Z)_{\text{TH}}$) can be estimated from the regression line equation as
191 function of the threshold $I_{\text{motor,TH}}$. Since, for the batch freezer used in the experiments, the producer
192 suggested $I_{\text{motor,TH}} = 6.5$ A, the corresponding thresholds for the electrical parameters are $|Z|_{\text{TH}} =$
193 755.3Ω , $\text{Arg}(Z)_{\text{TH}} = -13.58^\circ$ and $\Delta\text{Arg}(Z)_{\text{TH}} = 2.26^\circ$.

194 The repeatability of the experiments has been tested by means of 8 freezing assays performed under
195 the same conditions and the results, as well as statistics for the time to reach the stop process
196 threshold, are shown in Fig. 4. As can be seen, the freezing control using $|Z|$ leads to more
197 repeatable curves and minimum dispersion on the stopping time ($\Delta\text{time} = 1.85$ minutes), than those
198 exploiting measurements of $\text{Arg}(Z)$ ($\Delta\text{time} = 3.6$ minutes) and $\Delta\text{Arg}(Z)$ ($\Delta\text{time} = 3.1$ minutes).
199 These latter methods, however, exhibit smaller dispersion than straightforward I_{motor} control (Δtime
200 $= 3.88$ 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 $\text{Arg}(Z)$ significantly improves
204 the correlation ($R^2=0.79$ instead of $R^2=0.69$), while adding $\Delta\text{Arg}(Z)$ produces only a negligible
205 increase. Thus, in the following, only measurements of $|Z|$ and $\text{Arg}(Z)$ are considered.

206 The experiments described above were performed by cleaning the freezer with hot water before the
207 start of the process (so as to eliminate residuals of ice cream from previous assays and making the
208 initial temperature as homogeneous as possible) and filling the barrel always with the same quantity
209 of mix (corresponding to the maximum capacity of 7 liters). Since this is not the case during the
210 normal operation of a batch freezer, the effects of consecutive freezing processes without cleaning
211 as well as loading the freezer with different mix quantities have been studied.

212 To this purpose, three freezing procedures have been carried out in a row without cleaning the
213 machine barrel between consecutive assay and the results are presented in Fig. 5. As can be seen,
214 values of $|Z|$ are not significantly affected by the absence of barrel cleaning between different
215 freezing processes. On the contrary, $\text{Arg}(Z)$ exhibits significantly higher values only at the
216 beginning of the freezing process when the machine barrel is not cleaned from the previous
217 operation (possibly because of the presence of residuals of frozen ice cream between electrodes). As
218 freezing proceeds, however, these differences disappear and the data are no longer dependent on
219 repeated machine cleaning. Therefore, lack of cleaning between different freezings in practice does
220 not affect the reliability of electrical parameter measurements to determine the end of the freezing
221 process.

222 A typical situation where the freezing process fails to control the product stiffness is when the ice
223 cream freezes on the blades of the dasher motor, a problem more likely to occur when the cylinder
224 is fully loaded and the temperature is very low at the beginning of the process. To investigate the
225 capability of the type of control proposed in this work to overcome this problem, $|Z|$, $\text{Arg}(Z)$ and
226 I_{motor} have been monitored during freezing assays featuring ice cream freezing on the blades of the
227 dasher motor. The curves shown in Fig. 6 (a), (b) and (c) indicate that the values of I_{motor} remain
228 always lower than the threshold of 6.5 A, while $|Z|$ and $\text{Arg}(Z)$ reach their stop process thresholds
229 essentially at the same times than in the “standard” freezing experiments of Fig. 4. Therefore, the
230 combined monitor of both electrical parameters and I_{motor} can be used to detect the occurrence of the

231 problem of ice-cream freezing on the scraping blades (see Supplementary Material for $|Z|$ and
232 $\text{Arg}(Z)$ plotted vs. I_{motor}), allowing the user to promptly take countermeasures.

233 Fig. 7 (a), (b) and (c) show the time evolution of $|Z|$, $\text{Arg}(Z)$ 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
235 liters, 5 liters and 7 liters (maximum value of freezing capacity). Both $|Z|$ and I_{motor} do not reach the
236 corresponding stop process thresholds for the 3 liters case, while $\text{Arg}(Z)$ 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.

239 In particular, in the case of 3 liters, $|Z|$ initially increases (for the first 4 minutes), then slightly
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).

247 Even if measurements of neither $|Z|$ nor I_{motor} provide reliable control for the freezing process in the
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 $\text{Arg}(Z)$ 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)).

255 Denoting R_{AIR} and R_{MIX} the resistances of the air and the media, respectively, since $R_{\text{AIR}} \gg R_{\text{MIX}}$
256 the system impedance can be described as:

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

258 thus:

259 $|Z| = \sqrt{R_{MIX}^2 + \left(\frac{1}{\omega C_{MIX}}\right)^2}$, (2)

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

261 Denoting as R_{MIX}^* and C_{MIX}^* the resistance and capacitance for the electrodes covered with unit
 262 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:

264 $|Z| = \frac{1}{l} \cdot \sqrt{R_{MIX}^{*2} + \left(\frac{1}{\omega C_{MIX}^*}\right)^2}$, (4)

265 $Arg(Z) = -Arctg\left(\frac{1}{\omega R_{MIX}^* C_{MIX}^*}\right)$. (5)

266 Eq. 4 and 5 indicate that $|Z|$ decreases as l increase, while $Arg(Z)$ is independent of l .

267 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
 269 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
 272 composition and producers, and the results (presented in the Supplementary Material) are in good
 273 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.

276

277 **5. Conclusions**

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

288 The experimental data presented in the paper show a good linear correlation of both $|Z|$ and $\text{Arg}(Z)$
289 with the current drawn by the dasher motor, with $|Z|$ being the best choice as parameter to be
290 monitored because of higher data correlation and repeatability.

291 On the whole, the control of ice cream freezing process by electrical parameters monitoring
292 represents an economical and reliable technique advancing the state of the art in the field.

293

294 **Acknowledgements**

295 The authors would like to thank Carpigiani Group for providing the equipment used in the tests.

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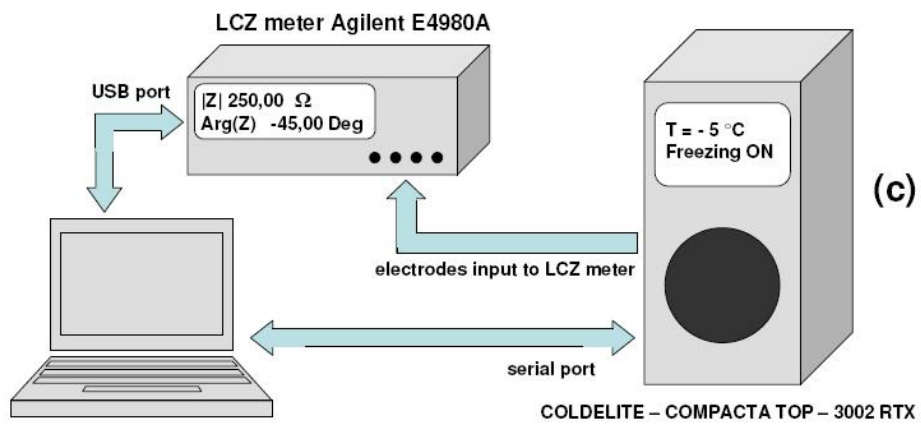
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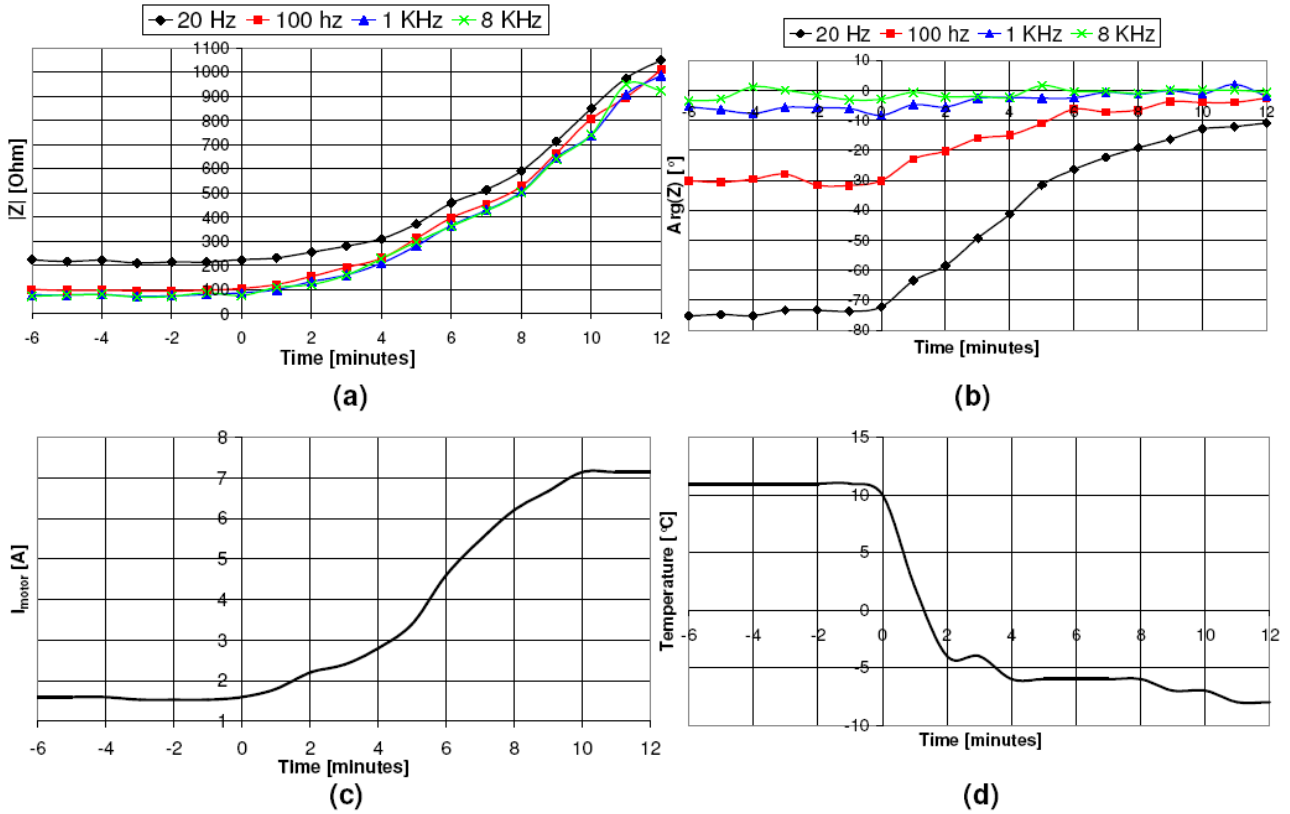
(b)



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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.

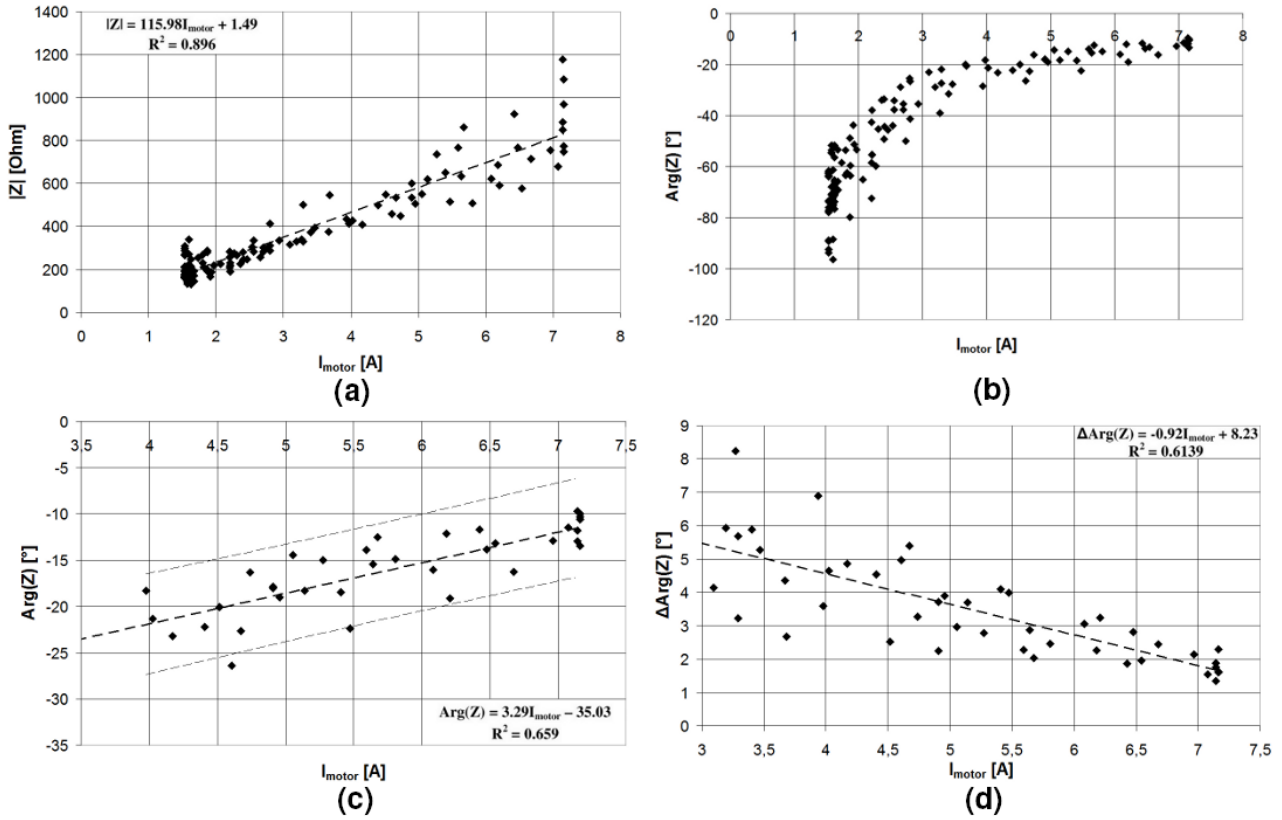
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Fig. 2 Curves of $|Z|$ and $\text{Arg}(Z)$ vs. time measured for different frequencies of the test signal (Fig. a and b, respectively). Current drawn by the dasher motor (c) and product temperature (d) as function of time during the freezing process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

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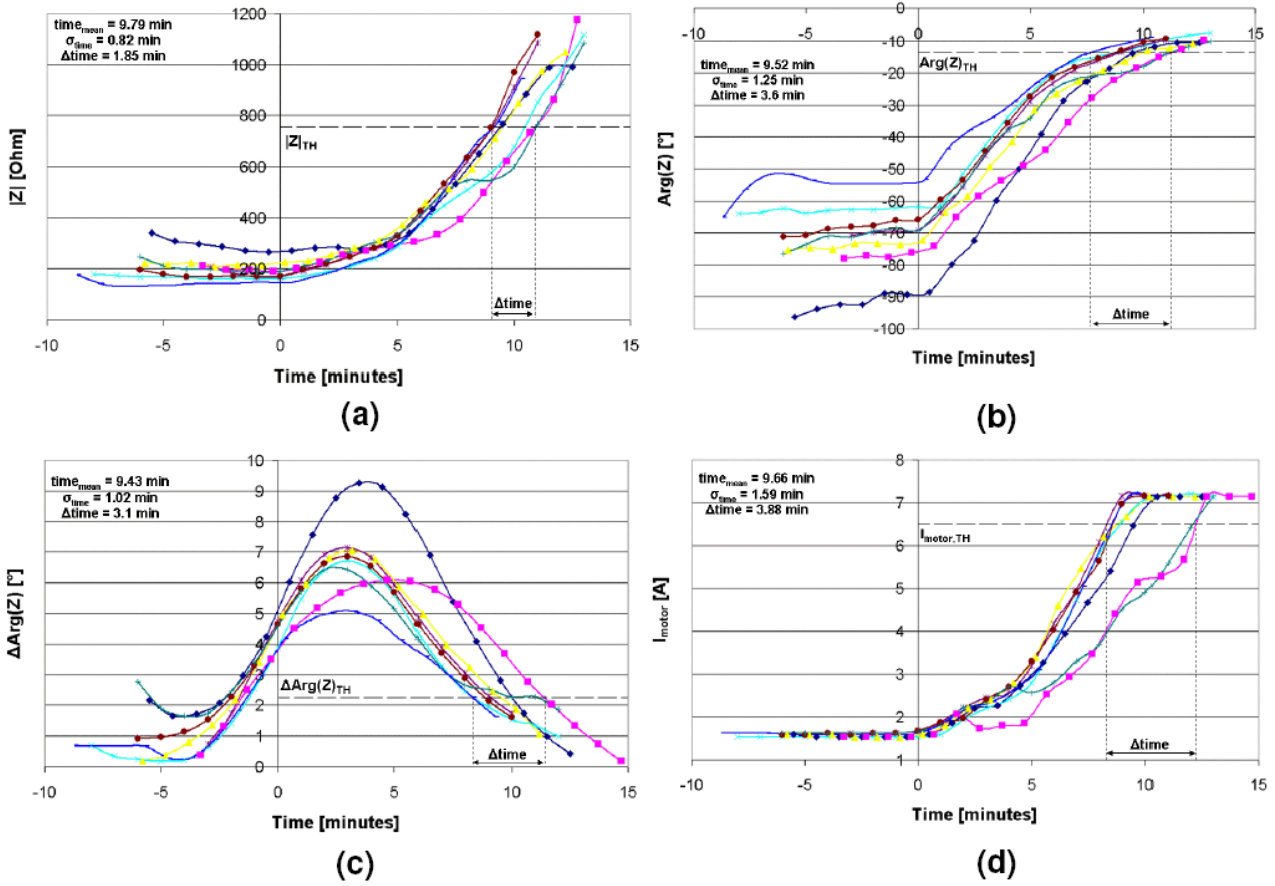


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439 **Fig. 3** Curves of $|Z|$ (a), $\text{Arg}(Z)$ and $\Delta\text{Arg}(Z)$ (d) vs. I_{motor} . Two curves $\text{Arg}(Z)$ vs. I_{motor} are
440 presented for the entire range values of I_{motor} (b) and for values in the range 3.5A to 7.5A (c).

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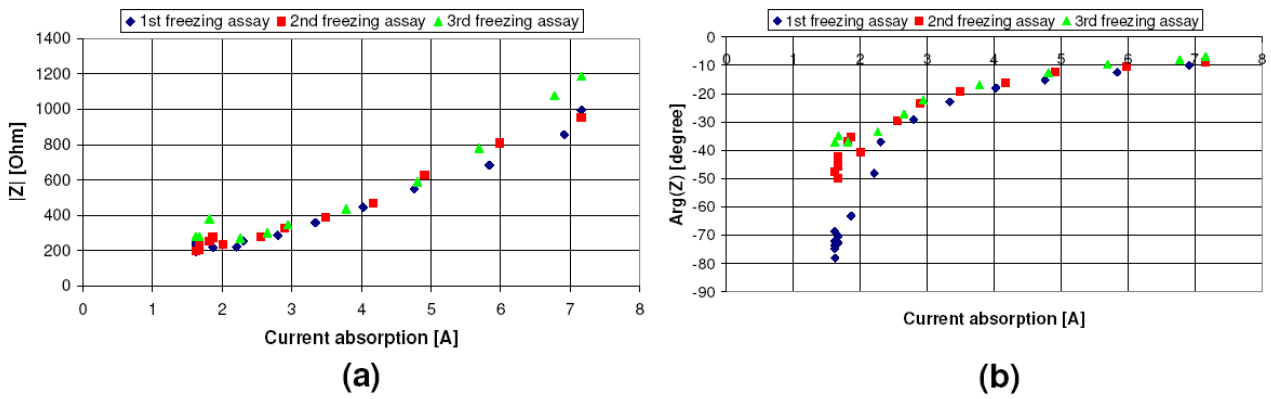
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 454 **Fig. 4** Repetition trials for the ice cream freezing process. The values of $|Z|$, $Arg(Z)$, $\Delta Arg(Z)$ and
 455 I_{motor} are plotted vs. time, while statistics for the stop process time are presented. The legend for the
 456 trial number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

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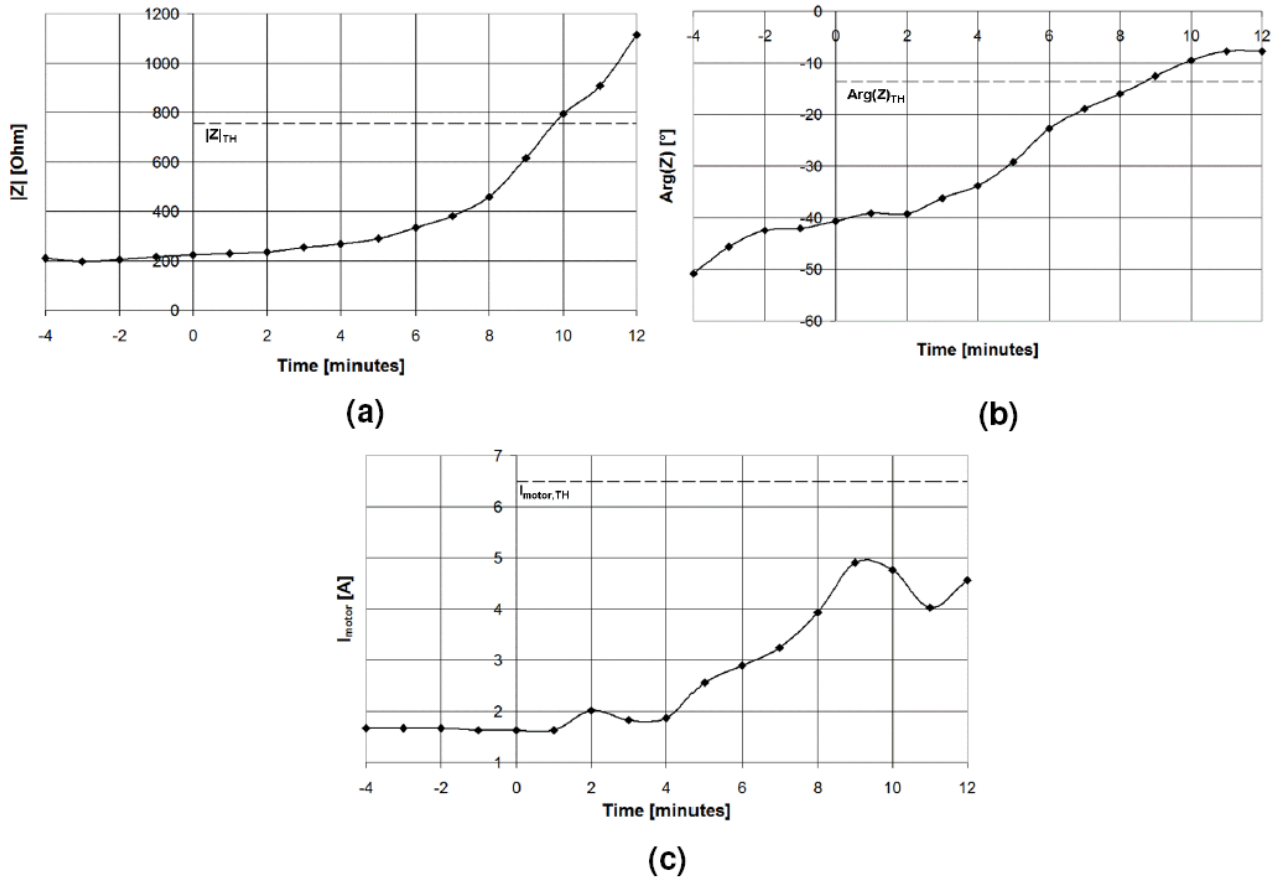
Fig. 5 $|Z|$ and $\text{Arg}(Z)$ vs. I_{motor} for freezing processes carried out without cleaning the machine barrel between consecutive assays.

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

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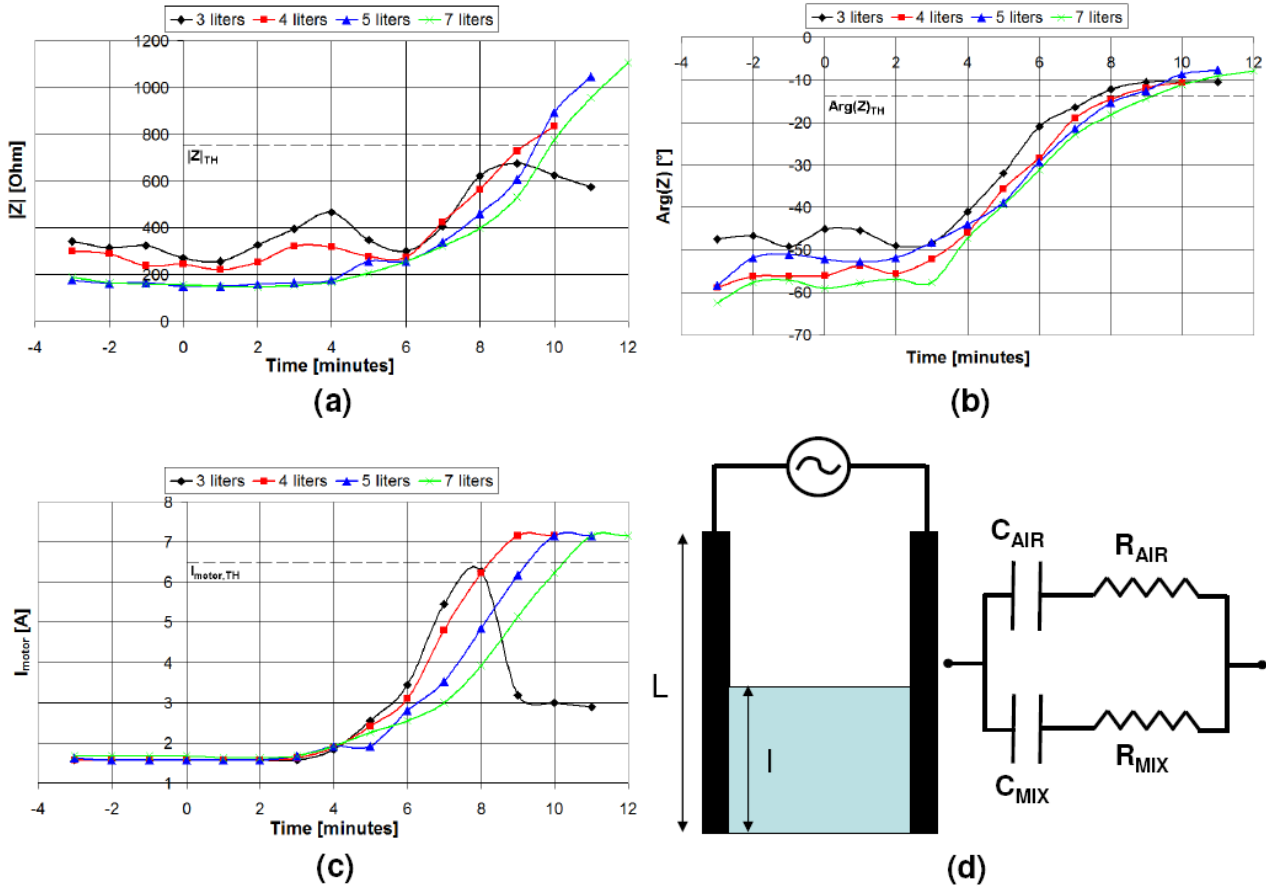
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502 **Fig. 7** Curves of $|Z|$, $Arg(Z)$ and I_{motor} vs. time for freezing processes carried out filling the machine

503 barrel with different volumes of ice cream mix (Fig. a, b and c, respectively). Electrical model for

504 the system formed by a couple of electrodes only partially covered by conductive media (d).

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Supplementary Material

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

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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 $\text{Arg}(Z)$ 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 at high frequency is significantly higher than at the beginning of the process.

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) has been used. Thus:

$$Z = R_{MIX} + \frac{1}{j\omega C_{MIX}} \quad \text{Re}(Z) = R_{MIX} \quad \text{Im}(Z) = -\frac{1}{\omega C_{MIX}}$$

This is consistent with Fig. S1 where $\text{Re}(Z)$ is essentially frequency independent and $\text{Im}(Z)$ decreases as the frequency increases.

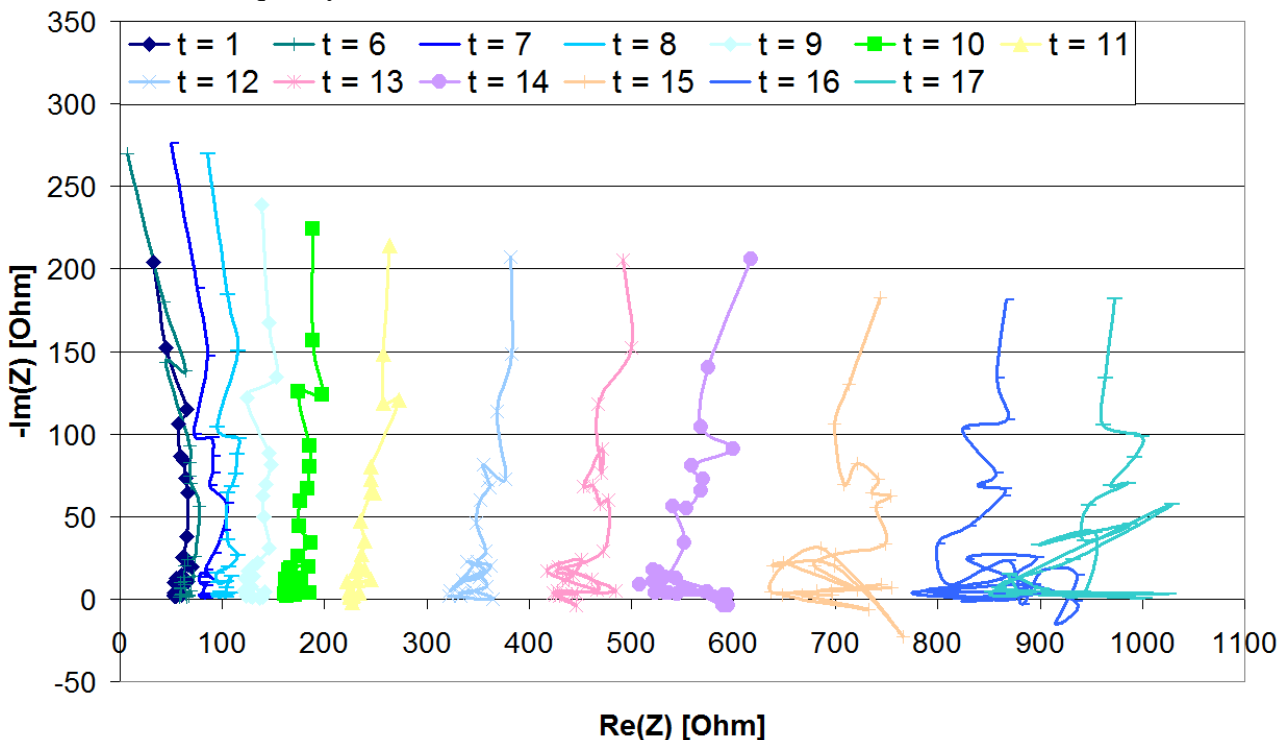
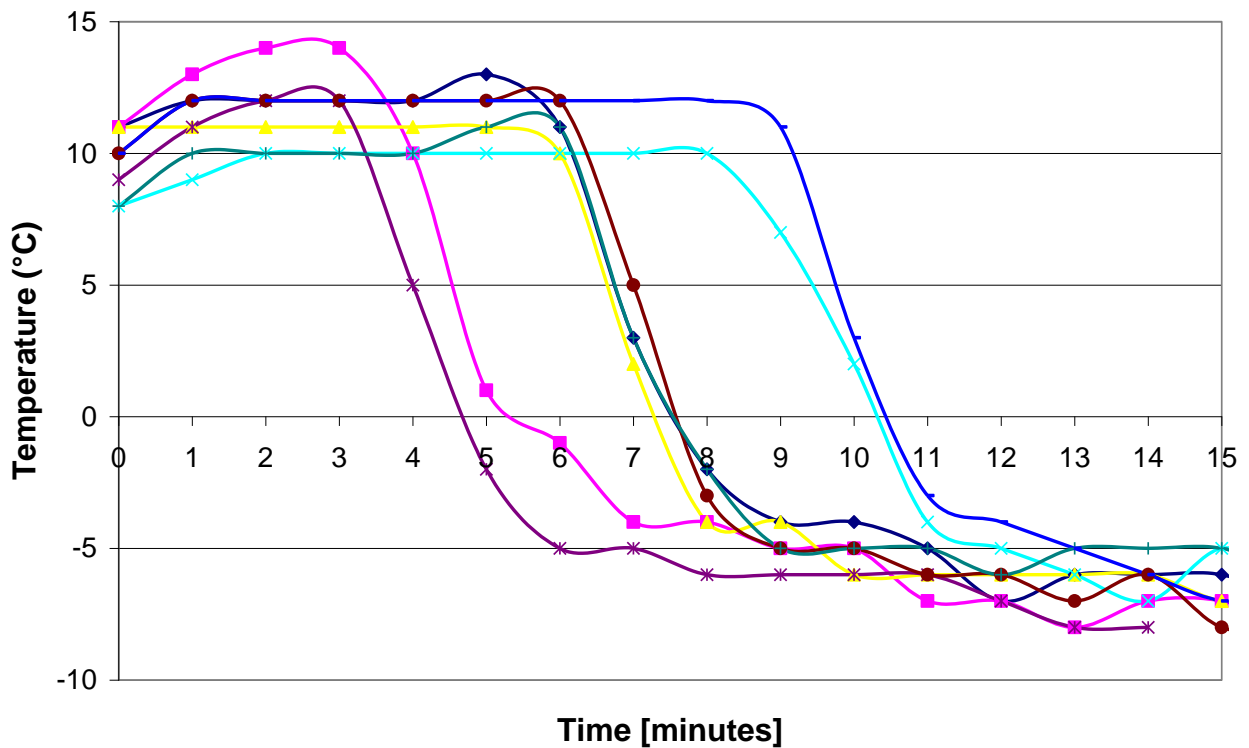


Fig. S1 Nyquist plots for different acquisition times (in minutes) during the freezing process.

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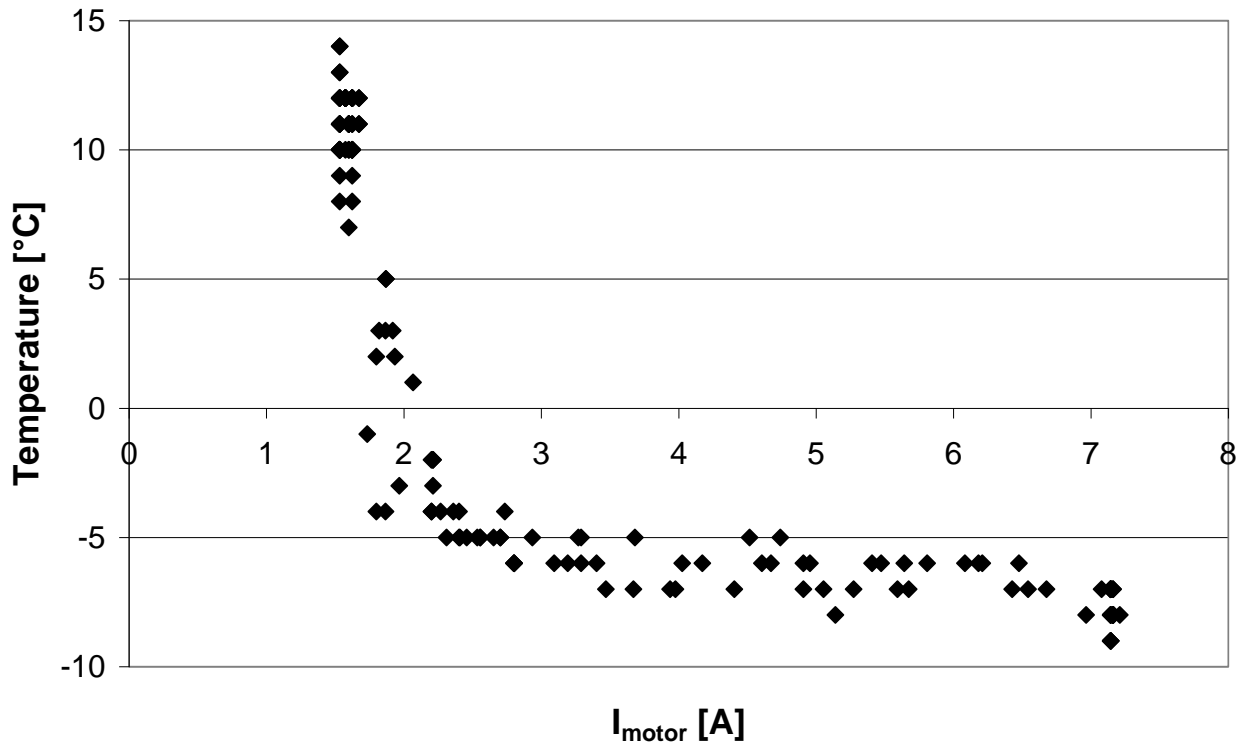


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552 **Fig. S2** Product temperature vs. acquisition time for different freezing assays.
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Correlation between product temperature and current drawn by the dasher motor. The product temperature is monitored 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 .

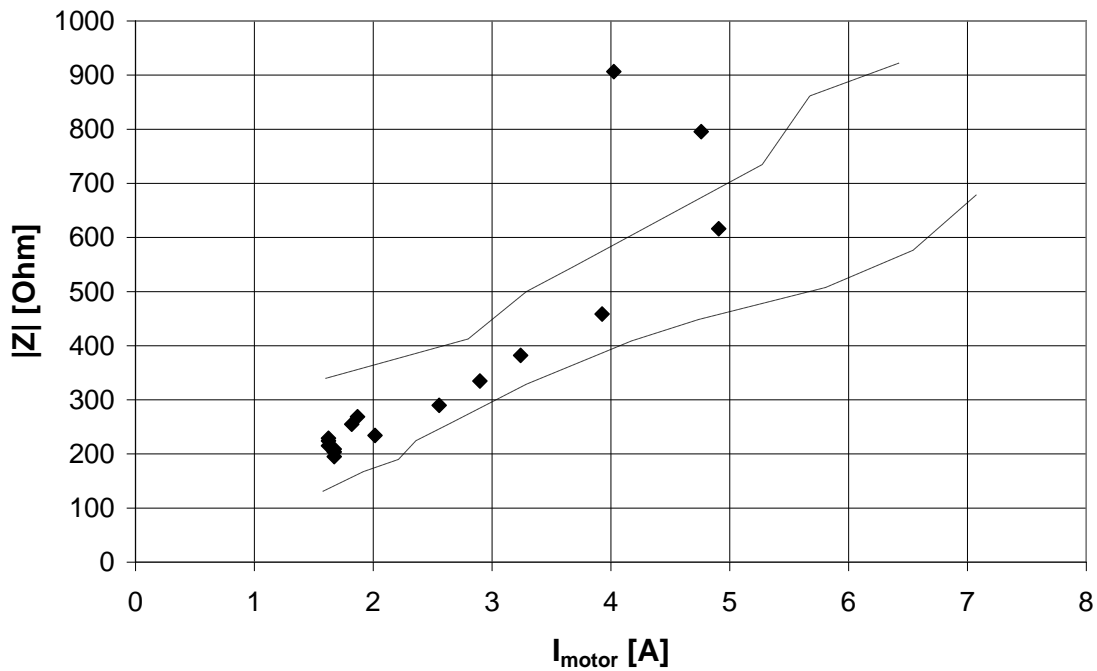


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580 **Fig. S3** Scatter plot for the product temperature vs. the current drawn by the dasher motor.
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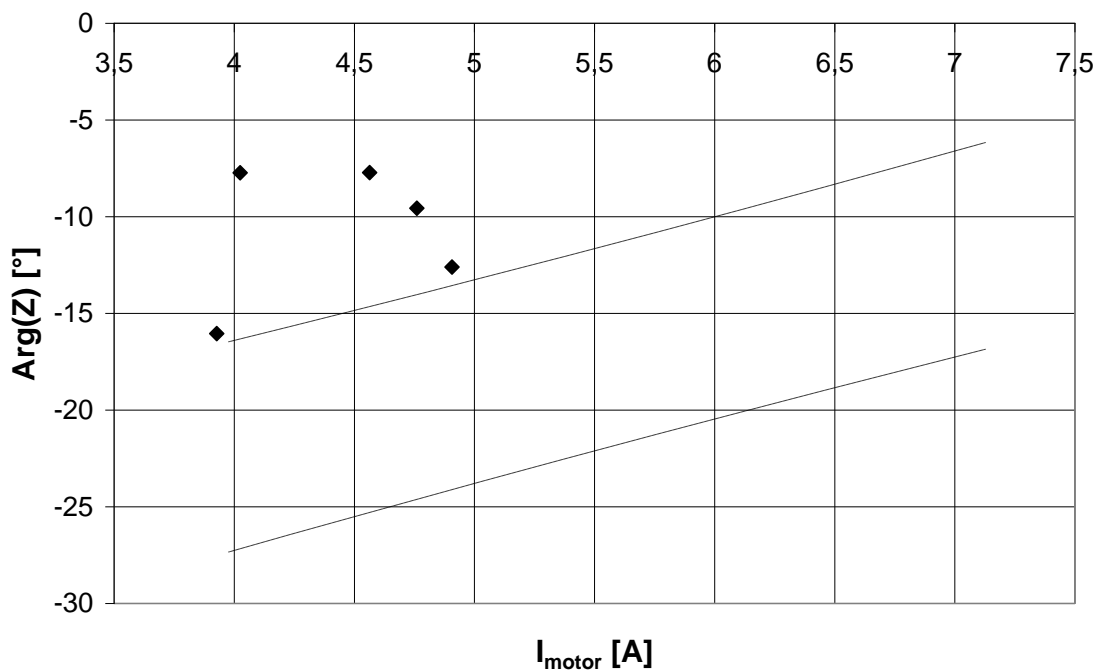
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601 Detection of ice cream mix freezing on the blades of the dasher motor by combined monitoring of
602 electrical characteristics and current drawn by the dasher motor. This problem can happen during
603 the freezing assay in particular when the freezer cylinder is loaded with maximum mix volume (7
604 liters) and the chamber temperature is very low already at the beginning of the process. This event
605 must be promptly detected since under this condition the proper control of product stiffness is
606 compromised. In Fig. S4 the monitored values for $|Z|$ and $\text{Arg}(Z)$ are plotted versus the
607 corresponding I_{motor} values for a freezing assay featuring this particular problem. Lower and higher
608 bounds from repeatability tests are also shown. As can be seen, as the mix freezes on the blades of
609 the dasher motor, values of I_{motor} decreases while $|Z|$ and $\text{Arg}(Z)$ increases. This causes the
610 measured data to fall off the safe working area represented by dashed lines in Fig. S4. Thus,
611 combined monitoring of $|Z|$, $\text{Arg}(Z)$ and I_{motor} can promptly detect this problem.



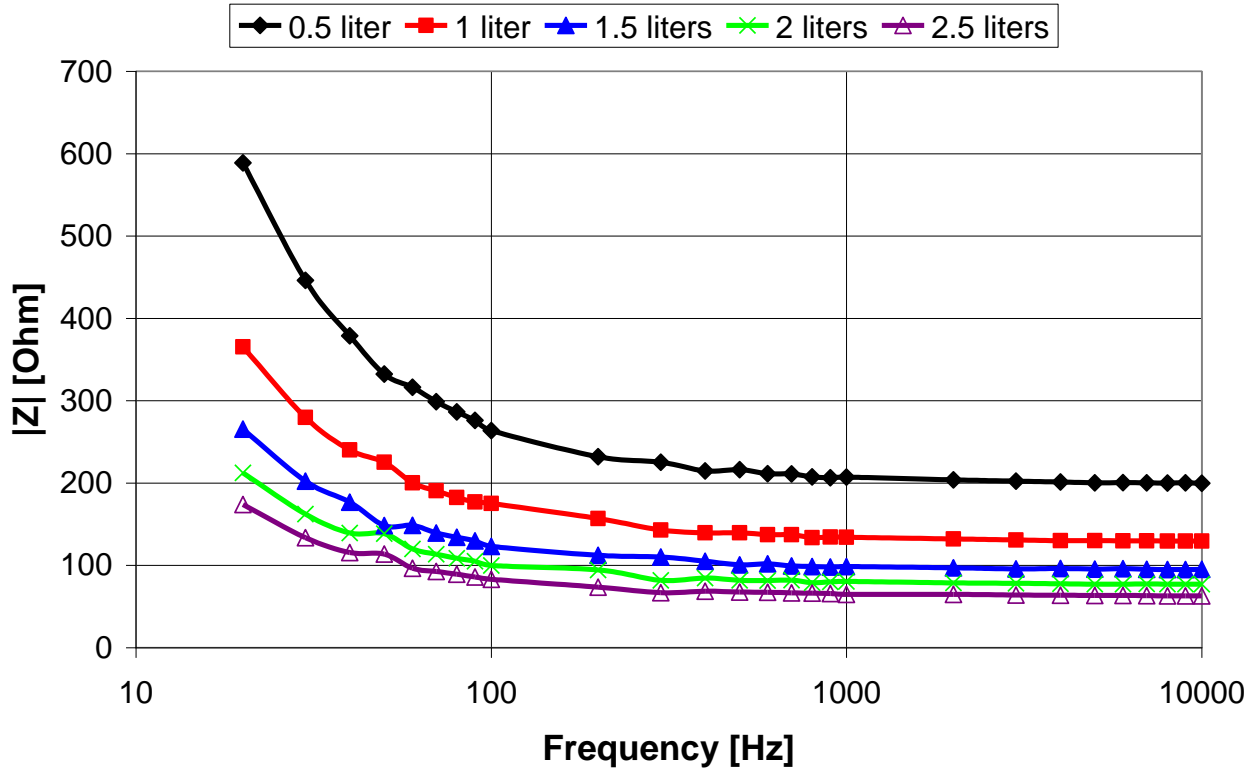
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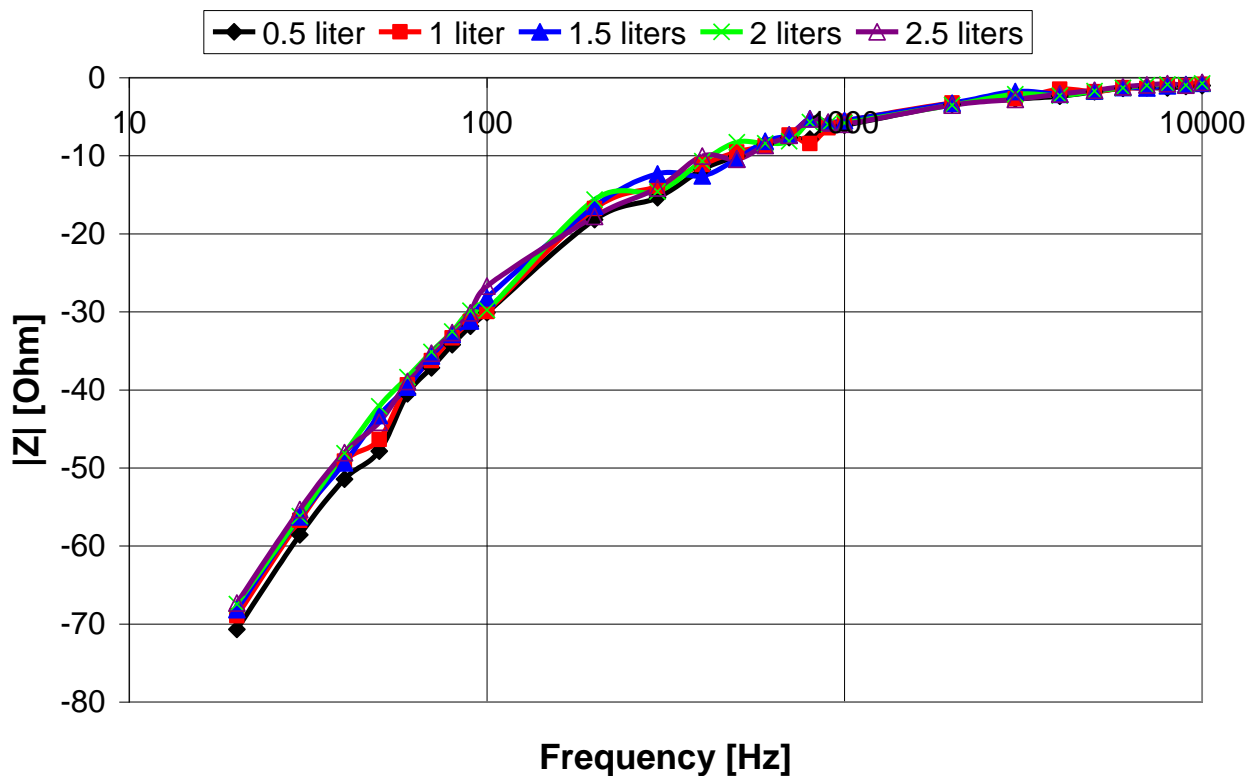
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614 **Fig. S4** Plots of $|Z|$ and $\text{Arg}(Z)$ vs. I_{motor} for a freezing assay featuring mix freezing on the blades of
615 the dasher motor.

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

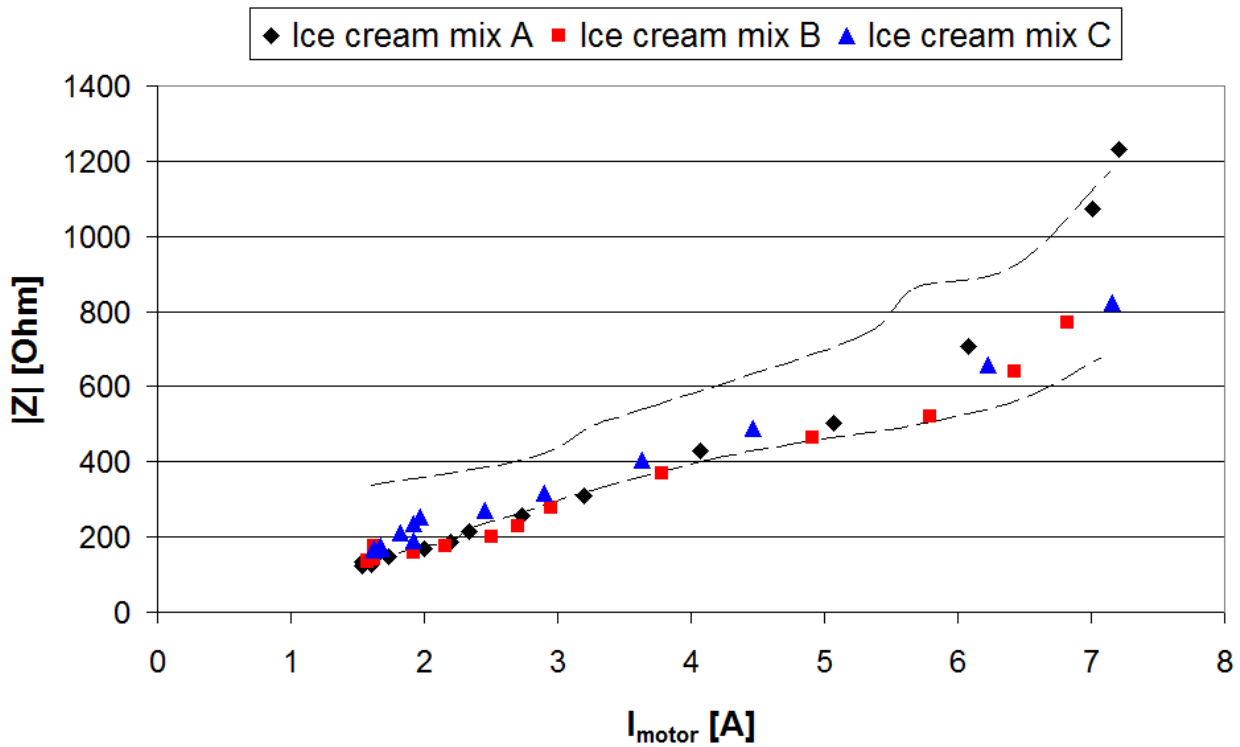


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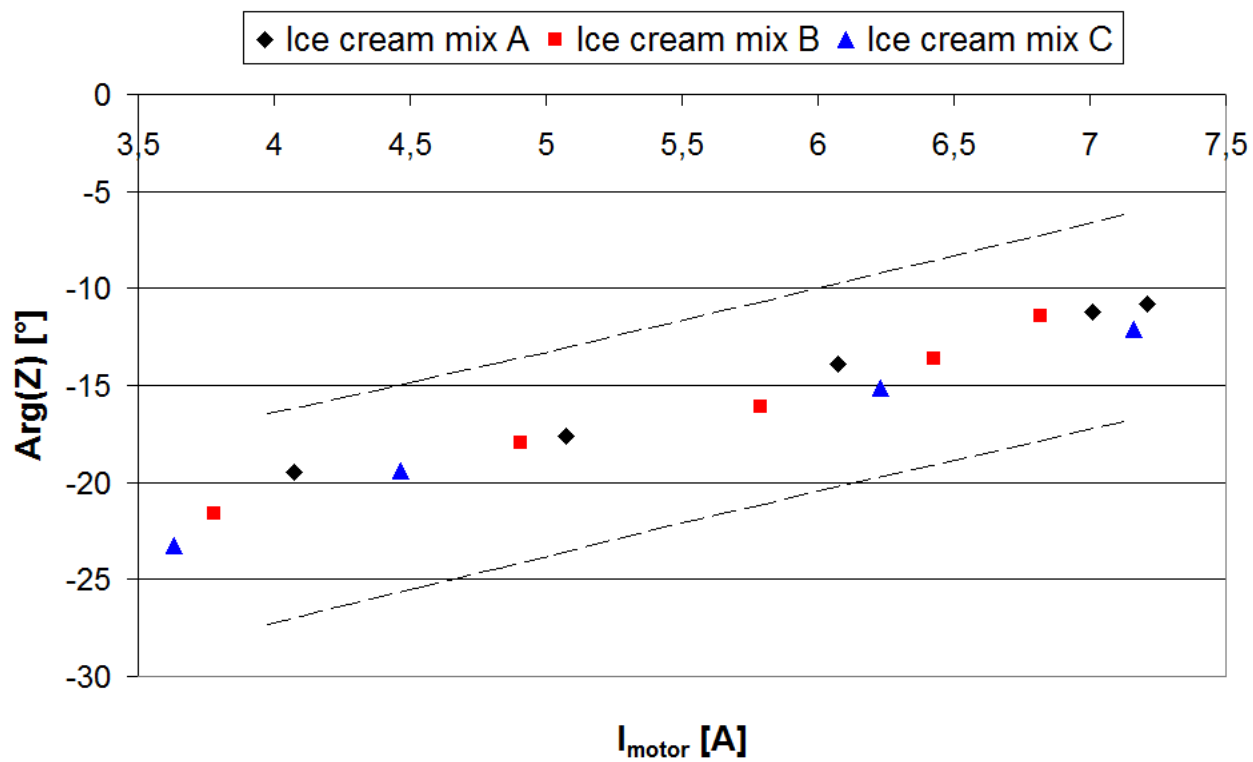


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624 **Fig. S5** $|Z|$ and $\text{Arg}(Z)$ as function of frequency for different ice cream mix volumes.
 625 *Measurements on different ice cream mixes.* Preliminary measurements have been carried out on a
 626 limited number of ice cream mix of different compositions and producers to test the correlation of
 627 the electrical parameters $|Z|$ and $\text{Arg}(Z)$ with the current drawn by the dasher motor and compare
 628 the results with those obtained with the test mix described in the Materials and Methods section.
 629 The recipes for the three product tested, hereafter named Ice Cream Mix A, B and C, are as follows.
 630
 631 Ice Cream Mix A: fat content 6%, milk solids-not-fat 12.5%, sugar 12%, corn syrup solids 4%,
 632 stabilizers/emulsifiers 0.4%, total solids 34.9%.
 633
 634 Ice Cream Mix B: fat content 10%, milk solids-not-fat 11%, sugar 12%, corn syrup solids 3%,
 635 stabilizers/emulsifiers 0.4%, total solids 36.4%.
 636
 637 Ice Cream Mix C: fat content 6%, milk solids-not-fat 11%, sugar 11%, corn syrup solids 5%,
 638 stabilizers/emulsifiers 0.5%, total solids 33.5%.
 639
 640 Ice Cream Mix A is produced by Berglandmilch (Austria, www.berglandmilch.at), while Ice Cream
 641 Mix B and C by Mondini nel Mondo (Rome, Italy, www.mondinimondo.it).
 642
 643 In Fig. S6 the electrical parameters $|Z|$ and $\text{Arg}(Z)$ are plotted vs current drawn by the dasher motor.
 644 Dashed lines represent the lower and higher bounds for the test mix described in the “Materials and
 645 Methods” section resulting from the scatter plots of Fig. 3 in the manuscript. As can be seen,
 646 measurements present good agreements with data obtained with the test mix, with only slightly
 647 lower values of $|Z|$ at the beginning of the freezing process.
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Fig. S6 Scatter plots for $|Z|$ and $\text{Arg}(Z)$ as function of the current drawn by the dasher motor for different ice cream mixes.