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22 **Abstract**

23

24 Recent studies have reported the use of microwave-induced combustion (MIC) for digestion
25 of several kinds of matrices. In spite of several applications of MIC, relatively few
26 information is available regarding the mechanism of ignition. In this work, a systematic study
27 related to the role of NH_4NO_3 solution and other nitrates for ignition step in MIC system was
28 performed. In this sense, aqueous solutions of $\text{Ca}(\text{NO}_3)_2$, KNO_3 , $\text{Mg}(\text{NO}_3)_2$, NaNO_3 and
29 NH_4NO_3 were evaluated. It was observed that the ignition is dependent on nitrate
30 concentration and microwave power and seems to be related to the oxidation of organic
31 matter by NO_3^- , which releases enough energy for starting a chain reaction leading to
32 combustion. Additionally, it was not observed a special action promoted by microwaves
33 without using nitrate solutions. According to the results, all the evaluated nitrate solutions can
34 be used as igniters with microwave power of 750 W or higher. It was also possible to use
35 nitrate solutions as diluted as 1 mol L^{-1} and relatively short time was required for ignition of
36 filter paper (below 10 s). Furthermore, the use of higher microwave power allowed a more
37 reproducible ignition.

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41

42 **Keywords:** Microwave-induced combustion; Ignition; Combustion Mechanism; Sample
43 preparation.

44

45 **Introduction**

46

47 Combustion is a very effective way to digest organic samples and it has been used for
48 sample digestion and subsequent determination of metals and non-metals by several analytical
49 techniques.¹⁻⁵ The efficiency of combustion is associated to the high temperature achieved
50 during sample burning that allows the decomposition of organic matrix, leading to a final
51 solution with low residual carbon content (RCC) that is fully compatible with many
52 determination techniques.² Combustion methods have been applied in open systems (e.g., dry
53 ashing, low temperature ashing and Wickbold combustion systems), closed systems (oxygen
54 flask and combustion bomb) and also in dynamic system (Trace-O-Mat).^{1,6} Ignition in closed
55 systems can be performed manually (oxygen flask method) but in general it requires a special
56 apparatus based on the application of an electrical current through a metal wire of Pt or Ni-Cr
57 (combustion bombs or oxygen flask) or using infrared lamps (oxygen flask). Despite the
58 spread use of these classical systems, some disadvantages can be pointed out regarding to
59 sample ignition. Although manual ignition is a simple way to start combustion (using oxygen
60 flask method), it is very dependent on the ability of analyst and could be unsuitable for routine
61 and safe operation.⁷ Ignition using electrical current has been used for combustion bombs, but
62 the use of metallic wires can be a potential contamination source because they are placed in
63 close contact with solid samples. On the other hand, infrared lamps avoid direct contact with
64 sample, but they require the use of vessels transparent to infrared radiation in order to allow
65 sample ignition, a special approach for the adjustment of lamp focus could be troublesome for
66 routine operation. In order to overcome these drawbacks related to sample ignition a system
67 was developed based on microwave radiation, called as microwave-induced combustion
68 (MIC) system.⁸

69 MIC system has been applied for the digestion of several kinds of samples,
70 particularly to those presenting organic matrix.^{3,9-18} In this method, sample combustion occurs
71 in open¹⁹⁻²¹ or closed vessels^{22,23} pressurized with oxygen with ignition started by microwave
72 radiation. Samples, pressed as pellets, are positioned on a quartz holder, placed inside the
73 vessel containing a small disc of filter paper wetted with a suitable igniter solution. After
74 combustion, products are absorbed in a convenient solution using a reflux step, which is
75 optional depending on the analyte. The applicability of MIC has been demonstrated for the
76 digestion of many matrices as well as for further determination of metals and non metals,
77 especially halogens.^{11,14} As observed for other combustion methods, MIC also allows the
78 digestion of samples that are considered difficult to bring into solution.¹⁰ Moreover, MIC
79 presents some practical benefits related to other combustion methods, such as higher sample
80 throughput and the application of a reflux step after combustion, improving analytes
81 recoveries. In addition, the use of diluted solutions for absorption of analytes allows
82 minimizing reagent consumption and waste generation.⁶

83 For MIC system, a 6 mol L⁻¹ NH₄NO₃ solution is typically used as igniter because this
84 reagent can be found in relatively high purity, it is relatively inexpensive and easily
85 commercially available. In addition, the products formed by NH₄NO₃ after combustion are
86 fully compatible with several determination techniques reducing the risks of interferences in
87 this step.^{6,7}

88 Although there are many applications of MIC for several matrices and analytes, there
89 is a lack of information regarding ignition mechanism and performance of other potential
90 solutions that could be used as igniters. It must be considered that the basic requirement for
91 ignition is an efficient energy transfer for the reactants, fuel (organic sample) and oxidant (air
92 or oxygen), to start the reaction, which proceeds smoothly to its completion up to a certain
93 critical limit. This limit is related to the rate of released energy that may exceed the rate of

94 energy lost to the vessel walls or gases by various processes of heat transfer. When this limit
95 is exceeded, reaction rate accelerates indefinitely leading to a fast self-acceleration of
96 reaction. Thus, this step where self-acceleration occurs is termed ignition.⁶

97 Up to now, the accepted mechanism of ignition in MIC system has been based on
98 empirical statements and suggests that microwave radiation induces a chemical reaction
99 between filter paper and NH_4NO_3 solution.⁶ Once the temperature of the whole system
100 (NH_4NO_3 solution and filter paper) presents a fast heating due to microwave absorption, it
101 causes a quick oxidation of the filter paper by NO_3^- . Considering a very localized heat that is
102 released by this reaction in an oxygen pressurized atmosphere, it provides enough energy for
103 sample ignition. However, a systematic study was never performed to confirm this hypothesis.

104 In the present work, several nitrate solutions were evaluated as igniters in order to
105 understand the ignition process in MIC system. The follow nitrate solutions were evaluated:
106 $\text{Ca}(\text{NO}_3)_2$, KNO_3 , $\text{Mg}(\text{NO}_3)_2$, NaNO_3 and also NH_4NO_3 . Furthermore, NH_4NO_3 solution was
107 used to evaluate the influence of microwave power delivered to the cavity on the ignition
108 process. Finally, it was also evaluated the effect of nitrate solutions for the combustion of
109 whole milk powder pellets leading to a better understanding of the microwave ignition using
110 nitrate solutions.

111

112

113 **Materials and methods**

114

115 **Instrumentation**

116

117 A microwave oven (Multiwave 3000 Sample Preparation System, Anton Paar, Graz,
118 Austria) equipped with up to eight high-pressure quartz vessels (internal volume of 80 mL,

119 maximum temperature and pressure of 280 °C and 80 bar, respectively) was used. A
120 commercial quartz holder (Anton Paar Cat. nr. 16427) was used to insert filter paper and
121 sample inside the quartz vessels. An analytical balance (model AY 220, max. 220 g, 0.0001 g
122 of resolution, Shimadzu, Kyoto, Japan) was used for weighing. A hydraulic press (Specac,
123 Orpington, UK) was used for the preparation of whole milk powder pellets. For both ignition
124 and combustion time evaluation, time was recorded by using a digital chronometer (Casio,
125 HS-3V-1BR, 10 ms of precision, Tokyo, Japan) and a digital video recording (Sony, DSC-
126 W800, 20.1 MP of resolution, Tokyo, Japan). A conventional oven with air circulation (model
127 400/2ND, Nova Ética, Brasil) was used for experiments performed with conventional heating.
128 A glass chamber (internal volume of 25 mL) was used for conventional heating experiments
129 under inert atmosphere (Ar).

130

131 **Reagents, samples and solutions**

132

133 Nitrate salts evaluated ($\text{Ca}(\text{NO}_3)_2$, KNO_3 , $\text{Mg}(\text{NO}_3)_2$, NaNO_3 and NH_4NO_3) were of
134 analytical grade (Merck, Darmstadt, Germany). Ultrapure water (Milli-Q system, 18.2 M Ω
135 cm) was used to prepare all solutions by direct dissolution of salts in water. Small discs of
136 cellulose filter paper (15 mm of diameter, 12 mg) with low ash content (Black Ribbon
137 Ashless, Schleicher and Schuell, Dassel, Germany) were used. Filter paper discs were
138 previously immersed in 5% HNO_3 solution by 20 min, washed thoroughly with water and
139 finally dried in a clean bench before use. Experiments were also carried out using whole milk
140 powder samples prepared as pellets (13 mm of diameter, pressed at 3 ton, 60 s). Oxygen
141 (99.6%, White Martins-Praxair, Brazil) was used for MIC method performed with microwave
142 heating, respectively. Argon (99.996%, White Martins-Praxair) was used for experiments
143 under inert atmosphere with both conventional and microwave heating.

144 **Evaluation of igniters solutions**

145

146 The solubility of each nitrate salt was considered for preparation of nitrate solutions at
147 variable concentrations (from 1 to 12 mol L⁻¹). A constant volume of 50 µL of each solution
148 was added on filter paper. Further, quartz holder was introduced into the quartz vessel
149 containing 6 mL of water as absorbing solution. Experiments were performed with filter paper
150 alone and also using filter paper plus whole milk powder pellet (100 mg). After closing the
151 rotor, vessels were pressurized with 20 bar of oxygen using the valve originally designed for
152 pressure release after conventional wet digestion. Further, rotor was inserted inside the oven
153 with the vessel positioned in front of the semi-transparent door. Maximum microwave power
154 (1400 W) was delivered during 60 s and immediately stopped just after ignition has been
155 started. A change in the original vessel protective case was performed to allow the
156 visualization of ignition and combustion times as shown in Figure 1. This change is not
157 recommended by microwave oven manufacturer. Therefore, for these tests, an acrylic wall (1
158 cm thickness) was adapted in front of microwave oven in order to provide a safer operation.

159 For safety, vessel was carefully opened for releasing the pressure only 5 min after the
160 end of combustion. For all experiments, each run was performed using four vessels in the
161 rotor.

162

163 **Figure 1**

164

165 **Results and discussion**

166

167 Ignition is the start point of combustion, which happens when the oxidation reaction is
168 accelerated and reagents are quickly consumed. During combustion, light emission is

169 generally observed and it is related to the temperature reached in this process. Therefore, a
170 visual evaluation of combustion is possible because a considerable part of radiation is emitted
171 in visible region due to the molecular transitions of flame products (H_2O , CO_2 , CO , O_2 , CH ,
172 etc.).^{2, 6, 24} In this way, the time required for ignition and also the combustion time were also
173 considered for evaluation of igniters.

174 Initial experiments were carried out without igniter addition using only filter paper and
175 whole milk powder with or without 50 μL of water under microwave heating up to 60 s
176 (Table 1).

177

178

Table 1

179

180 In the experiments performed with dry filter paper alone or with sample, as well as
181 those performed with the addition of 50 μL of water on the filter paper, it was not observed
182 any indication related to the beginning of combustion or even the oxidation of filter paper or
183 sample. In the same way, the presence of sample did not change these results. In these
184 experiments, both filter paper and sample remained unchanged after microwave irradiation
185 showing that there is no specific microwave effect on filter paper or sample in order to allow
186 ignition (Figure 2). As expected, these results shown that only the interaction of microwaves
187 with filter paper or sample was not enough to start the combustion and the addition of an
188 igniter solution combined to microwave irradiation is necessary for ignition.

189

190

Figure 2

191

192 The use of ammonium nitrate in solid state was also evaluated under microwave
193 heating (20 bar of oxygen, one filter paper and around 10 mg salt). Two experiments were

194 performed: *i*) NH_4NO_3 salt previously dried in an oven (100 °C); and, *ii*) NH_4NO_3 salt with
195 "natural" moisture (this reagent has a hygroscopic nature and presented around 3% of water in
196 this experiment). Using NH_4NO_3 (previously dried) no combustion or even oxidation of filter
197 paper was observed (Figure 3A). However, when NH_4NO_3 with natural moisture was used the
198 filter paper was partially burned (Figure 3B), being an indicative about the importance of
199 water in oxidation process.

200

201 **Figure 3**

202

203 **Evaluation of nitrate solutions as igniters**

204

205 Solutions of $\text{Ca}(\text{NO}_3)_2$, KNO_3 , $\text{Mg}(\text{NO}_3)_2$ and NaNO_3 were initially evaluated using
206 only filter paper. Initially, solutions of these salts were arbitrarily selected as 5, 3, 3 and 7 mol
207 L^{-1} , respectively and results are shown in Table 2.

208

209 **Table 2**

210

211 It was observed that filter paper burned when every nitrate solution was added,
212 showing the possibility of using not only NH_4NO_3 (as in the previous works using MIC) but
213 also other nitrate salts as igniters. This result also reinforces the importance of the oxidation
214 of organic matter (starting from filter paper) by nitrates allowing ignition. Since all evaluated
215 nitrate salts resulted in paper ignition, a systematic study was performed in order to evaluate
216 the influence of concentration of each one that should be enough to allow paper combustion.
217 Results are shown in Table 3.

218

219

Table 3

220

221 All evaluated concentrations of nitrate solutions resulted in paper ignition. The final
222 aspect of quartz holder after filter paper combustion showed some dark parts (Figure 4) that
223 were not observed when sample was placed together the filter paper. It is due to the relatively
224 lower heat released during combustion (low amount of organic matter in contact with the cold
225 surface of the quartz holder) and the absence of reflux step of absorbing solution after
226 combustion. When MIC method is used with sample (even up to 500 mg), the final aspect of
227 quartz holder after combustion is as a clear surface.

228

229

Figure 4

230

231 As shown in Table 3, in general the higher the concentration of nitrate solution the
232 shorter the time required for ignition. It can be explained by the fast oxidation of filter paper
233 caused by the increase of nitrate concentration. This effect is better observed for $\text{Ca}(\text{NO}_3)_2$
234 and NH_4NO_3 solutions, where the time observed for ignition decreases more than three
235 seconds. The same behavior was not observed for NaNO_3 . For this reagent, the evaluation of
236 the ignition time resulted in large standard deviation for all concentrations caused by the
237 incostant burning of filter paper when this solution was used.

238

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243

Although the ignition time is dependent on the nitrate concentration, no significant
difference was observed in the time of combustion for solutions of the same salt (ANOVA,
confidence level of 95%), because the mass of filter paper (12 mg) and O_2 pressure (20 bar)
were kept constant and after ignition no changes in combustion process were expected.
Moreover, it was showed that the kind of salt did not affect the combustion of filter paper,
which reinforces the statement related to the oxidation action promoted by NO_3^- mainly for

244 ignition step. Although the differences of ignition time, it is important to point out that even
245 using nitrate solutions as diluted as 1 mol L^{-1} the combustion of filter paper was possible.

246

247 **Evaluation of oxidation promoted by nitrate solutions**

248 In order to evaluate the oxidation of paper by NO_3^- , filter papers were wetted with 50
249 μL of 7 mol L^{-1} NH_4NO_3 solution and further introduced inside a conventional oven heated at
250 $170 \text{ }^\circ\text{C}$. In such conditions, it was observed the darkening of filter paper (Figure 5A), being an
251 indicative of oxidation reaction. For assuring that oxidation reaction was not caused by the
252 action of oxygen present in air, filter paper wetted with ammonium nitrate was positioned
253 inside a closed glass chamber, previously purged with Ar (inert atmosphere). As presented in
254 Figure 5B, even in inert atmosphere a darkening of filter paper was observed, which
255 reinforces the hypothesis of paper oxidation caused mainly due to NO_3^- . Additionally, a
256 similar experiment was performed by using microwave heating under an inert atmosphere
257 (quartz vessel purged with Ar). As shown in Figure 5C, the darkening of filter paper was
258 again observed.

259

260

260 **Figure 5**

261

262 Therefore, it was observed that filter paper becomes dark in both conventional and
263 microwave heating. Although no ignition was observed in these experiments, probably due to
264 the lower heating rate observed in conventional oven or due to the absence of oxygen (oven
265 and microwave heating under inert atmosphere), the darkening of filter paper can be related to
266 the oxidant action of NO_3^- added to the paper.

267

268 **The effect of microwave power on the ignition process**

269

270 In order to evaluate the effect of microwave power on the ignition process, the
271 microwave power delivered into cavity was studied from 250 to 1400 W (using 7 mol L⁻¹
272 NH₄NO₃ as combustion igniter) as shown in Table 4.

273

274 **Table 4**

275

276 It was observed in Table 4 that when low microwave power was used (750 W or less),
277 the ignition of filter paper was delayed and presented higher standard deviation (about 25%).
278 In addition, when 250 and 500 W were used the combustion of filter paper was not observed
279 in some cases, leading to a non reproducible combustion. On the other hand, the use of higher
280 microwave power provides a faster ignition of filter paper, as it was observed when 1000 and
281 1400 W were selected. Additionally, it was also observed a better repeatability of experiments
282 when higher microwave power was used. The heating process must be fast enough for
283 initiating a chain reaction responsible for ignition. If these reactions are faster the heat
284 released by filter paper will not be lost to the holder surface or oxygen gas, being enough to
285 allow the ignition.

286

287 **Igniters evaluation in presence of sample pellet**

288

289 After the evaluation of several nitrate solutions for combustion of filter paper it was
290 investigated their use for the digestion of whole milk powder. The igniter (50 µL) was added
291 on filter paper and sample prepared as pellet (100 mg) was positioned on this paper. Results
292 are shown in Table 5.

293

Table 5

294

295 All nitrate solutions evaluated were considered as suitable for ignition. The ignition
296 and combustion times were very similar, as well as the deviation among replicates showing
297 that other nitrate salts could be used instead NH_4NO_3 for MIC. Moreover, the possibility of
298 using several nitrate solutions, which represents an alternative for sample preparation by MIC,
299 was considered as valuable for better understanding the mechanism of combustion started by
300 microwave radiation. However, considering the applications using MIC digestion, it is
301 necessary to point out that depending on the selected igniter some elements could be not
302 determined in digests such as Ca, K, Mg and Na if they are present in selected igniter salt.
303 This aspect must be considered before using any alternative igniter nitrate aiming the
304 determination of these elements.

305

306 Conclusions and novelty features

307

308 When microwave radiation was applied to dried paper or with water, combustion was
309 not observed indicating that nitrate solutions were important for sample ignition in MIC.
310 Independent of the kind of nitrate salt used, it was possible to observe the ignition for all the
311 nitrate solutions investigated in this work. Water presented an important role in this process
312 and no oxidation of filter paper was observed by using dried solid NH_4NO_3 . On the other
313 hand, the atmosphere inside combustion vessel did not change the reaction behavior
314 indicating the oxidation of paper by NO_3^- as an important reaction related to the mechanism of
315 ignition.

316

317 Considering the solutions used for MIC, it was normally observed a faster ignition
when more concentrated nitrate solutions were used, which was related to the effect caused by

318 a faster oxidation of the filter paper. The same behaviour was observed by increasing the
319 microwave power. Additionally, it was also observed that the use of higher microwave power
320 (750 W or higher) was necessary in order to ensure a reproducible combustion process. On
321 the other hand, the time observed during combustion was almost the same for several igniter
322 concentrations, which was expected once the paper mass and concentration of oxygen were
323 kept constant for all evaluations.

324 In this way, microwave-induced combustion can be performed using other nitrate salts
325 in addition to NH_4NO_3 making the couple of microwave heating and combustion a versatile
326 alternative for achieving effective fast ignition.

327

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329

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332

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334

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390 **Figure captions**

391

392 **Figure 1.** Front view of digestion vessel with a window which was opened in the protective
393 case used for ignition and combustion time evaluation.

394

395 **Figure 2.** Aspect of filter paper wetted with 50 μL of water (A) and sample positioned on the
396 filter paper previously wetted with 50 μL of water (B) after microwave irradiation
397 ($n = 3$). Experiments were performed using 6 mL of H_2O as absorbing solution;
398 microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.

399

400 **Figure 3.** Evaluation of NH_4NO_3 salt used as igniter (solid state) after microwave irradiation.
401 (A) final aspect of filter paper using NH_4NO_3 dried in conventional oven. (B) final
402 aspect of filter paper using NH_4NO_3 with natural moisture (3% of hygroscopic
403 moisture). Experiments were performed using 10 mg of NH_4NO_3 salt; microwave
404 power of 1400 W up to 60 s; 20 bar of oxygen pressure.

405

406 **Figure 4.** Final aspect of quartz holder after filter paper combustion when diluted solution of
407 ammonium nitrate (7 mol L^{-1}) was as igniter. Combustion performed using 6 mL of
408 H_2O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of
409 oxygen pressure ($n = 3$).

410

411 **Figure 5.** Evaluation of oxidation action of NH_4NO_3 solution over filter paper: (A) aspect of
412 filter paper after heating at 170 $^\circ\text{C}$ in an oven (air atmosphere, around 20% of O_2);

413 (B) aspect of filter paper after heating at 170 °C in an oven with inert atmosphere
414 (glass chamber purged with Ar); (C) aspect of filter paper after microwave
415 irradiation under inert atmosphere (vessel purged with Ar). Experiments were
416 performed using 50 µL of 7 mol L⁻¹ NH₄NO₃ solution as igniter; microwave power
417 of 1400 W up to 60 s; 20 bar of oxygen pressure.

418

419 **Table 1.** Evaluation of the effect of microwaves without addition of nitrate solutions on the
420 combustion of filter paper and whole milk powder sample (n = 3).

Experiments	Dry filter paper (12 mg)	Water addition to filter paper (50 μ L)	Sample (100 mg)	Observed combustion
1	Yes	No	No	No
2	Yes	No	Yes	No
3	Yes	Yes	No	No
4	Yes	Yes	Yes	No

421 Experiments performed using 6 mL of H₂O as absorbing solution; microwave power of 1400 W up to
422 60 s; 20 bar of oxygen pressure.
423

424 **Table 2.** Evaluation of different nitrate solutions used for combustion of filter paper (n = 3).

Nitrate solutions	Concentration, mol L ⁻¹	Observed combustion
Ca(NO ₃) ₂	5	Yes
KNO ₃	3	Yes
Mg(NO ₃) ₂	3	Yes
NaNO ₃	7	Yes

425 Combustion performed using 6 mL of H₂O as absorbing solution; microwave power of 1400 W up to
426 60 s; 20 bar of oxygen pressure.
427

428 **Table 3.** Evaluation of combustion of filter paper (without sample) with different concentrations of
 429 nitrate solutions (50 μL) ($n = 3$).

Igniter solution	Concentration, mol L^{-1}	Observed combustion	Ignition time, s	Combustion time, s
$\text{Ca}(\text{NO}_3)_2$	1	Yes	8.0 ± 1.0	7.9 ± 1.0
	3	Yes	5.3 ± 1.2	7.7 ± 1.5
	5	Yes	4.7 ± 0.6	7.0 ± 1.0
KNO_3	1	Yes	8.7 ± 1.2	7.0 ± 1.0
	3	Yes	8.0 ± 1.0	5.7 ± 1.5
$\text{Mg}(\text{NO}_3)_2$	1	Yes	6.0 ± 2.0	6.7 ± 2.1
	3	Yes	5.3 ± 1.5	7.7 ± 4.7
NaNO_3	1	Yes	8.0 ± 3.6	6.7 ± 1.5
	3	Yes	6.0 ± 3.0	5.7 ± 2.3
	5	Yes	5.7 ± 2.9	6.0 ± 1.0
	7	Yes	5.7 ± 1.2	6.3 ± 0.6
NH_4NO_3	1	Yes	7.3 ± 1.5	6.0 ± 1.7
	3	Yes	6.3 ± 2.0	6.3 ± 1.2
	5	Yes	5.3 ± 1.5	7.0 ± 1.0
	7	Yes	4.7 ± 1.5	6.7 ± 0.6
	9	Yes	4.7 ± 0.6	6.3 ± 0.6
	12	Yes	4.3 ± 0.6	6.0 ± 1.0

430 Combustion performed using 6 mL of H_2O as absorbing solution; microwave power of 1400 W up to
 431 60 s; 20 bar of oxygen pressure.
 432

433 **Table 4.** Evaluation of the effect of microwave power on the combustion of filter paper using 7 mol L⁻¹
434 NH₄NO₃ as igniter.

Microwave Power, W	Observed combustion (replicates)			Ignition time, s	Combustion time, s
	Experiment 1	Experiment 2	Experiment 3		
250	No	No	Yes	55.0	7.0
500	Yes	Yes	No	18.5 ± 4.9	6.7 ± 1.5
750	Yes	Yes	Yes	21.0 ± 5.0	6.9 ± 1.4
1000	Yes	Yes	Yes	5.3 ± 1.5	7.3 ± 1.2
1400	Yes	Yes	Yes	4.7 ± 1.2	7.0 ± 1.0

435 Combustion performed using 6 mL of H₂O as absorbing solution; microwave irradiation up to 60 s; 20
436 bar of oxygen pressure.
437

438 **Table 5.** Evaluation of different nitrate solutions for combustion of whole milk powder (100 mg) (n =
439 3).

Igniter solution	Concentration, mol L ⁻¹	Observed combustion	Ignition time, s	Combustion time, s
Ca(NO ₃) ₂	5	Yes	6.5 ± 0.5	5.5 ± 0.5
KNO ₃	3	Yes	5.5 ± 0.5	7.5 ± 0.5
Mg(NO ₃) ₂	3	Yes	6.5 ± 0.5	6.5 ± 0.5
NaNO ₃	7	Yes	5.0 ± 1.0	6.0 ± 1.0
NH ₄ NO ₃	12	Yes	6.7 ± 0.6	7.5 ± 0.5

440 Combustion performed using 6 mL of H₂O as absorbing solution; microwave power of 1400 W up to
441 60 s; 20 bar of oxygen pressure.
442

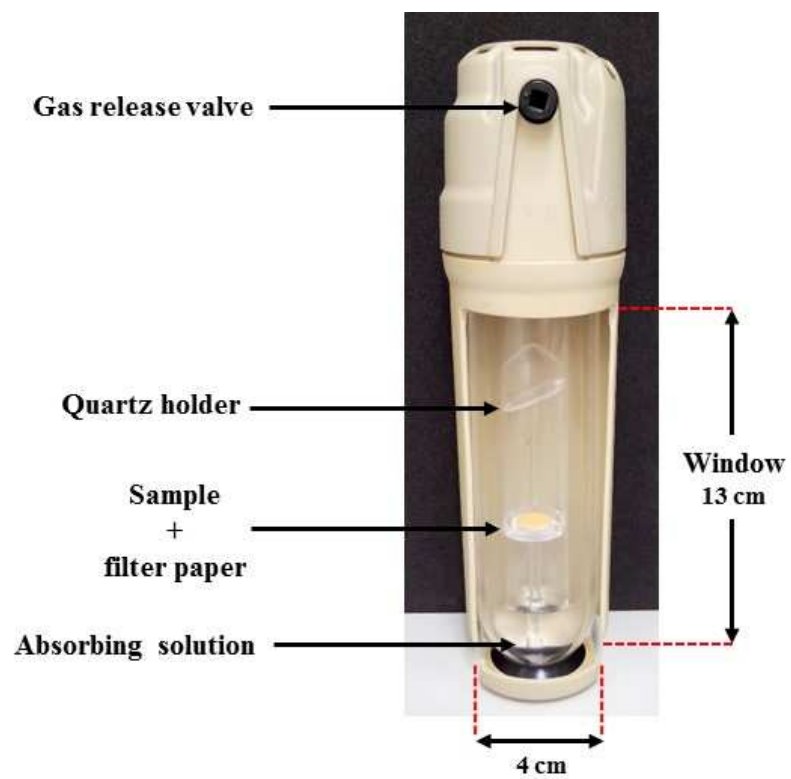
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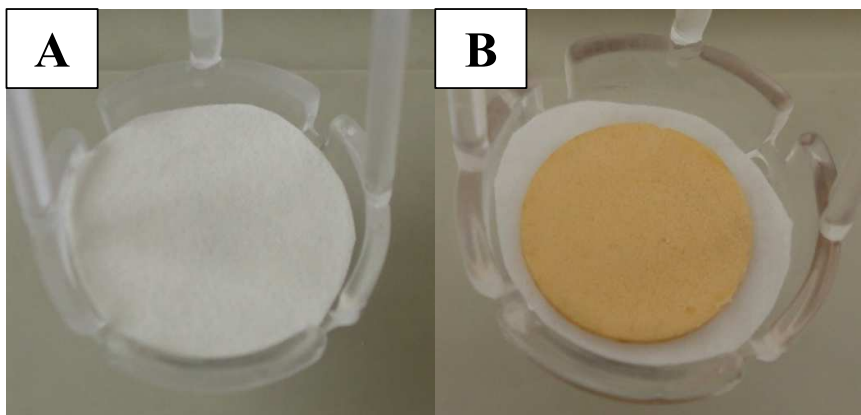
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446

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449 **Figure 1**

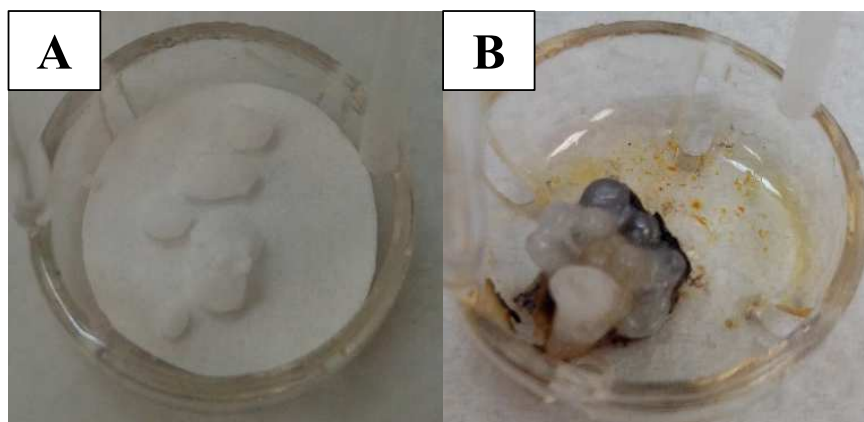
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451

452 **Figure 2**

453



454

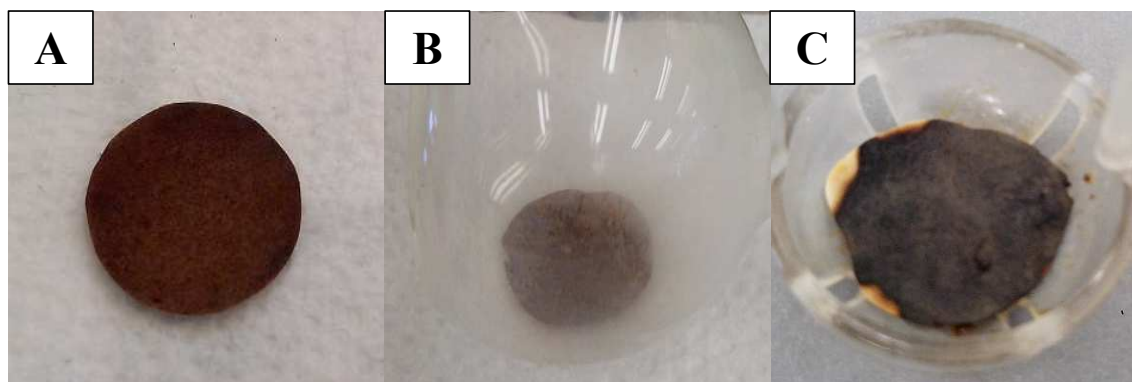
455 **Figure 3**

456



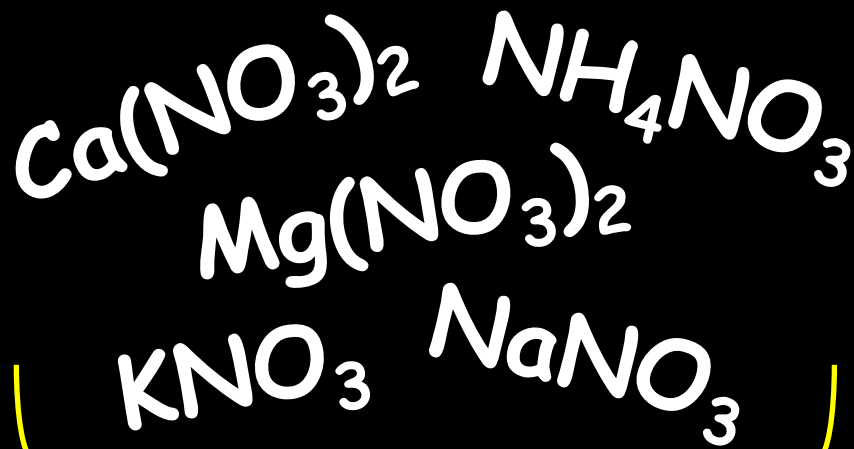
458 **Figure 4**

459



460

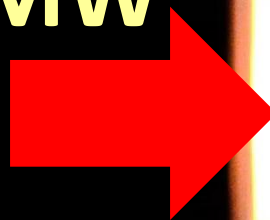
461 **Figure 5**



IGNITER



MW



COMBUSTION