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Evaluation of Nitrates as Igniters for Microwave-Induced Combustion: Understanding the Mechanism of Ignition

Leticia S. F. Pereira\textsuperscript{a}, Cezar A. Bizzi\textsuperscript{a}, Lucas Schmidt\textsuperscript{a}, Marcia F. Mesko\textsuperscript{b}, Juliano S. Barin\textsuperscript{c}, Erico M. M. Flores\textsuperscript{a,}\textsuperscript{*}

\textsuperscript{a} Departamento de Química, Universidade Federal de Santa Maria, 97105-900, Santa Maria, RS, Brazil

\textsuperscript{b} Centro de Ciências Químicas, Farmacêuticas e de Alimentos, Universidade Federal de Pelotas, 96010-610, Pelotas, RS, Brazil

\textsuperscript{c} Departamento de Tecnologia e Ciência de Alimentos, Universidade Federal de Santa Maria, 97105-900, Santa Maria, RS, Brazil

*Corresponding author: Tel/Fax: +55-55-3220-9445

E-mail address: (Érico M. M. Flores): ericommf@gmail.com
Abstract

Recent studies have reported the use of microwave-induced combustion (MIC) for digestion of several kinds of matrices. In spite of several applications of MIC, relatively few information is available regarding the mechanism of ignition. In this work, a systematic study related to the role of NH$_4$NO$_3$ solution and other nitrates for ignition step in MIC system was performed. In this sense, aqueous solutions of Ca(NO$_3$)$_2$, KNO$_3$, Mg(NO$_3$)$_2$, NaNO$_3$ and NH$_4$NO$_3$ were evaluated. It was observed that the ignition is dependent on nitrate concentration and microwave power and seems to be related to the oxidation of organic matter by NO$_3^-$, which releases enough energy for starting a chain reaction leading to combustion. Additionally, it was not observed a special action promoted by microwaves without using nitrate solutions. According to the results, all the evaluated nitrate solutions can be used as igniters with microwave power of 750 W or higher. It was also possible to use nitrate solutions as diluted as 1 mol L$^{-1}$ and relatively short time was required for ignition of filter paper (below 10 s). Furthermore, the use of higher microwave power allowed a more reproducible ignition.

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

Combustion is a very effective way to digest organic samples and it has been used for sample digestion and subsequent determination of metals and non-metals by several analytical techniques.\(^1\text{-}^5\) The efficiency of combustion is associated to the high temperature achieved during sample burning that allows the decomposition of organic matrix, leading to a final solution with low residual carbon content (RCC) that is fully compatible with many determination techniques.\(^2\) Combustion methods have been applied in open systems (e.g., dry ashing, low temperature ashing and Wickbold combustion systems), closed systems (oxygen flask and combustion bomb) and also in dynamic system (Trace-O-Mat).\(^1,^6\) Ignition in closed systems can be performed manually (oxygen flask method) but in general it requires a special apparatus based on the application of an electrical current through a metal wire of Pt or Ni-Cr (combustion bombs or oxygen flask) or using infrared lamps (oxygen flask). Despite the spread use of these classical systems, some disadvantages can be pointed out regarding to sample ignition. Although manual ignition is a simple way to start combustion (using oxygen flask method), it is very dependent on the ability of analyst and could be unsuitable for routine and safe operation.\(^7\) Ignition using electrical current has been used for combustion bombs, but the use of metallic wires can be a potential contamination source because they are placed in close contact with solid samples. On the other hand, infrared lamps avoid direct contact with sample, but they require the use of vessels transparent to infrared radiation in order to allow sample ignition, a special approach for the adjustment of lamp focus could be troublesome for routine operation. In order to overcome these drawbacks related to sample ignition a system was developed based on microwave radiation, called as microwave-induced combustion (MIC) system.\(^8\)
MIC system has been applied for the digestion of several kinds of samples, particularly to those presenting organic matrix. In this method, sample combustion occurs in open or closed vessels pressurized with oxygen with ignition started by microwave radiation. Samples, pressed as pellets, are positioned on a quartz holder, placed inside the vessel containing a small disc of filter paper wetted with a suitable igniter solution. After combustion, products are absorbed in a convenient solution using a reflux step, which is optional depending on the analyte. The applicability of MIC has been demonstrated for the digestion of many matrices as well as for further determination of metals and non-metals, especially halogens. As observed for other combustion methods, MIC also allows the digestion of samples that are considered difficult to bring into solution. Moreover, MIC presents some practical benefits related to other combustion methods, such as higher sample throughput and the application of a reflux step after combustion, improving analytes recoveries. In addition, the use of diluted solutions for absorption of analytes allows minimizing reagent consumption and waste generation.

For MIC system, a 6 mol L\(^{-1}\) \(\text{NH}_4\text{NO}_3\) solution is typically used as igniter because this reagent can be found in relatively high purity, it is relatively inexpensive and easily commercially available. In addition, the products formed by \(\text{NH}_4\text{NO}_3\) after combustion are fully compatible with several determination techniques reducing the risks of interferences in this step.

Although there are many applications of MIC for several matrices and analytes, there is a lack of information regarding ignition mechanism and performance of other potential solutions that could be used as igniters. It must be considered that the basic requirement for ignition is an efficient energy transfer for the reactants, fuel (organic sample) and oxidant (air or oxygen), to start the reaction, which proceeds smoothly to its completion up to a certain critical limit. This limit is related to the rate of released energy that may exceed the rate of
energy lost to the vessel walls or gases by various processes of heat transfer. When this limit is exceeded, reaction rate accelerates indefinitely leading to a fast self-acceleration of reaction. Thus, this step where self-acceleration occurs is termed ignition.\textsuperscript{6}

Up to now, the accepted mechanism of ignition in MIC system has been based on empirical statements and suggests that microwave radiation induces a chemical reaction between filter paper and NH\textsubscript{4}NO\textsubscript{3} solution.\textsuperscript{6} Once the temperature of the whole system (NH\textsubscript{4}NO\textsubscript{3} solution and filter paper) presents a fast heating due to microwave absorption, it causes a quick oxidation of the filter paper by NO\textsubscript{3}\textsuperscript{-}. Considering a very localized heat that is released by this reaction in an oxygen pressurized atmosphere, it provides enough energy for sample ignition. However, a systematic study was never performed to confirm this hypothesis.

In the present work, several nitrate solutions were evaluated as igniters in order to understand the ignition process in MIC system. The follow nitrate solutions were evaluated: Ca(NO\textsubscript{3})\textsubscript{2}, KNO\textsubscript{3}, Mg(NO\textsubscript{3})\textsubscript{2} NaNO\textsubscript{3} and also NH\textsubscript{4}NO\textsubscript{3}. Furthermore, NH\textsubscript{4}NO\textsubscript{3} solution was used to evaluate the influence of microwave power delivered to the cavity on the ignition process. Finally, it was also evaluated the effect of nitrate solutions for the combustion of whole milk powder pellets leading to a better understanding of the microwave ignition using nitrate solutions.

Materials and methods

Instrumentation

A microwave oven (Multiwave 3000 Sample Preparation System, Anton Paar, Graz, Austria) equipped with up to eight high-pressure quartz vessels (internal volume of 80 mL,
maximum temperature and pressure of 280 °C and 80 bar, respectively) was used. A commercial quartz holder (Anton Paar Cat. nr. 16427) was used to insert filter paper and sample inside the quartz vessels. An analytical balance (model AY 220, max. 220 g, 0.0001 g of resolution, Shimadzu, Kyoto, Japan) was used for weighing. A hydraulic press (Specac, Orpington, UK) was used for the preparation of whole milk powder pellets. For both ignition and combustion time evaluation, time was recorded by using a digital chronometer (Casio, HS-3V-1BR, 10 ms of precision, Tokyo, Japan) and a digital video recording (Sony, DSC-W800, 20.1 MP of resolution, Tokyo, Japan). A conventional oven with air circulation (model 400/2ND, Nova Ética, Brasil) was used for experiments performed with conventional heating. A glass chamber (internal volume of 25 mL) was used for conventional heating experiments under inert atmosphere (Ar).

**Reagents, samples and solutions**

Nitrate salts evaluated (Ca(NO$_3$)$_2$, KNO$_3$, Mg(NO$_3$)$_2$, NaNO$_3$ and NH$_4$NO$_3$) were of analytical grade (Merck, Darmstadt, Germany). Ultrapure water (Milli-Q system, 18.2 MΩ cm) was used to prepare all solutions by direct dissolution of salts in water. Small discs of cellulose filter paper (15 mm of diameter, 12 mg) with low ash content (Black Ribbon Ashless, Schleicher and Schuell, Dassel, Germany) were used. Filter paper discs were previously immersed in 5% HNO$_3$ solution by 20 min, washed thoroughly with water and finally dried in a clean bench before use. Experiments were also carried out using whole milk powder samples prepared as pellets (13 mm of diameter, pressed at 3 ton, 60 s). Oxygen (99.6%, White Martins-Praxair, Brazil) was used for MIC method performed with microwave heating, respectively. Argon (99.996%, White Martins-Praxair) was used for experiments under inert atmosphere with both conventional and microwave heating.
Evaluation of igniters solutions

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

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

Figure 1

Results and discussion

Ignition is the start point of combustion, which happens when the oxidation reaction is accelerated and reagents are quickly consumed. During combustion, light emission is
generally observed and it is related to the temperature reached in this process. Therefore, a visual evaluation of combustion is possible because a considerable part of radiation is emitted in visible region due to the molecular transitions of flame products (H\textsubscript{2}O, CO\textsubscript{2}, CO, O\textsubscript{2}, CH, etc.).\textsuperscript{2, 6, 24} In this way, the time required for ignition and also the combustion time were also considered for evaluation of igniters.

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

**Table 1**

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

**Figure 2**

The use of ammonium nitrate in solid state was also evaluated under microwave heating (20 bar of oxygen, one filter paper and around 10 mg salt). Two experiments were
performed: *i*) NH₄NO₃ salt previously dried in an oven (100 °C); and, *ii*) NH₄NO₃ salt with "natural" moisture (this reagent has a hygroscopic nature and presented around 3% of water in this experiment). Using NH₄NO₃ (previously dried) no combustion or even oxidation of filter paper was observed (Figure 3A). However, when NH₄NO₃ with natural moisture was used the filter paper was partially burned (Figure 3B), being an indicative about the importance of water in oxidation process.

**Figure 3**

**Evaluation of nitrate solutions as igniters**

Solutions of Ca(NO₃)₂, KNO₃, Mg(NO₃)₂ and NaNO₃ were initially evaluated using only filter paper. Initially, solutions of these salts were arbitrarily selected as 5, 3, 3 and 7 mol L⁻¹, respectively and results are shown in Table 2.

**Table 2**

It was observed that filter paper burned when every nitrate solution was added, showing the possibility of using not only NH₄NO₃ (as in the previous works using MIC) but also other nitrate salts as igniters. This result also reinforces the importance of the oxidation of organic matter (starting from filter paper) by nitrates allowing ignition. Since all evaluated nitrate salts resulted in paper ignition, a systematic study was performed in order to evaluate the influence of concentration of each one that should be enough to allow paper combustion. Results are shown in Table 3.
All evaluated concentrations of nitrate solutions resulted in paper ignition. The final aspect of quartz holder after filter paper combustion showed some dark parts (Figure 4) that were not observed when sample was placed together the filter paper. It is due to the relatively lower heat released during combustion (low amount of organic matter in contact with the cold surface of the quartz holder) and the absence of reflux step of absorbing solution after combustion. When MIC method is used with sample (even up to 500 mg), the final aspect of quartz holder after combustion is as a clear surface.

As shown in Table 3, in general the higher the concentration of nitrate solution the shorter the time required for ignition. It can be explained by the fast oxidation of filter paper caused by the increase of nitrate concentration. This effect is better observed for Ca(NO$_3$)$_2$ and NH$_4$NO$_3$ solutions, where the time observed for ignition decreases more than three seconds. The same behavior was not observed for NaNO$_3$. For this reagent, the evaluation of the ignition time resulted in large standard deviation for all concentrations caused by the incostant burning of filter paper when this solution was used.

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

**Evaluation of oxidation promoted by nitrate solutions**

In order to evaluate the oxidation of paper by NO\(_3^-\), filter papers were wetted with 50 \(\mu\)L of 7 mol L\(^{-1}\) NH\(_4\)NO\(_3\) solution and further introduced inside a conventional oven heated at 170 °C. In such conditions, it was observed the darkening of filter paper (Figure 5A), being an indicative of oxidation reaction. For assuring that oxidation reaction was not caused by the action of oxygen present in air, filter paper wetted with ammonium nitrate was positioned inside a closed glass chamber, previously purged with Ar (inert atmosphere). As presented in Figure 5B, even in inert atmosphere a darkening of filter paper was observed, which reinforces the hypothesis of paper oxidation caused mainly due to NO\(_3^-\). Additionally, a similar experiment was performed by using microwave heating under an inert atmosphere (quartz vessel purged with Ar). As shown in Figure 5C, the darkening of filter paper was again observed.

**Figure 5**

Therefore, it was observed that filter paper becomes dark in both conventional and microwave heating. Although no ignition was observed in these experiments, probably due to the lower heating rate observed in conventional oven or due to the absence of oxygen (oven and microwave heating under inert atmosphere), the darkening of filter paper can be related to the oxidant action of NO\(_3^-\) added to the paper.
The effect of microwave power on the ignition process

In order to evaluate the effect of microwave power on the ignition process, the microwave power delivered into cavity was studied from 250 to 1400 W (using 7 mol L\(^{-1}\) NH\(_4\)NO\(_3\) as combustion igniter) as shown in Table 4.

Table 4

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

Igniters evaluation in presence of sample pellet

After the evaluation of several nitrate solutions for combustion of filter paper it was investigated their use for the digestion of whole milk powder. The igniter (50 µL) was added on filter paper and sample prepared as pellet (100 mg) was positioned on this paper. Results are shown in Table 5.
All nitrate solutions evaluated were considered as suitable for ignition. The ignition and combustion times were very similar, as well as the deviation among replicates showing that other nitrate salts could be used instead NH\textsubscript{4}NO\textsubscript{3} for MIC. Moreover, the possibility of using several nitrate solutions, which represents an alternative for sample preparation by MIC, was considered as valuable for better understanding the mechanism of combustion started by microwave radiation. However, considering the applications using MIC digestion, it is necessary to point out that depending on the selected igniter some elements could be not determined in digests such as Ca, K, Mg and Na if they are present in selected igniter salt. This aspect must be considered before using any alternative igniter nitrate aiming the determination of these elements.

**Conclusions and novelty features**

When microwave radiation was applied to dried paper or with water, combustion was not observed indicating that nitrate solutions were important for sample ignition in MIC. Independent of the kind of nitrate salt used, it was possible to observe the ignition for all the nitrate solutions investigated in this work. Water presented an important role in this process and no oxidation of filter paper was observed by using dried solid NH\textsubscript{4}NO\textsubscript{3}. On the other hand, the atmosphere inside combustion vessel did not change the reaction behavior indicating the oxidation of paper by NO\textsubscript{3}\textsuperscript{-} as an important reaction related to the mechanism of ignition.

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

In this way, microwave-induced combustion can be performed using other nitrate salts in addition to NH$_4$NO$_3$ making the couple of microwave heating and combustion a versatile alternative for achieving effective fast ignition.
Acknowledgements

The authors are grateful to FAPERGS, INCT-Bioanalítica, CNPq and CAPES for supporting this study.
References


Figure captions

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

**Figure 2.** Aspect of filter paper wetted with 50 µL of water (A) and sample positioned on the filter paper previously wetted with 50 µL of water (B) after microwave irradiation (n = 3). Experiments were performed using 6 mL of H$_2$O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.

**Figure 3.** Evaluation of NH$_4$NO$_3$ salt used as igniter (solid state) after microwave irradiation. (A) final aspect of filter paper using NH$_4$NO$_3$ dried in conventional oven. (B) final aspect of filter paper using NH$_4$NO$_3$ with natural moisture (3% of hygroscopic moisture). Experiments were performed using 10 mg of NH$_4$NO$_3$ salt; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.

**Figure 4.** Final aspect of quartz holder after filter paper combustion when diluted solution of ammonium nitrate (7 mol L$^{-1}$) was as igniter. Combustion performed using 6 mL of H$_2$O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure (n = 3).

**Figure 5.** Evaluation of oxidation action of NH$_4$NO$_3$ solution over filter paper: (A) aspect of filter paper after heating at 170 ºC in an oven (air atmosphere, around 20% of O$_2$);
(B) aspect of filter paper after heating at 170 °C in an oven with inert atmosphere (glass chamber purged with Ar); (C) aspect of filter paper after microwave irradiation under inert atmosphere (vessel purged with Ar). Experiments were performed using 50 µL of 7 mol L\(^{-1}\) NH\(_4\)NO\(_3\) solution as igniter; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.
Table 1. Evaluation of the effect of microwaves without addition of nitrate solutions on the combustion of filter paper and whole milk powder sample (n = 3).

<table>
<thead>
<tr>
<th>Experiments</th>
<th>Dry filter paper (12 mg)</th>
<th>Water addition to filter paper (50 µL)</th>
<th>Sample (100 mg)</th>
<th>Observed combustion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>2</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>4</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
</tbody>
</table>

Experiments performed using 6 mL of H₂O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.
Table 2. Evaluation of different nitrate solutions used for combustion of filter paper (n = 3).

<table>
<thead>
<tr>
<th>Nitrate solutions</th>
<th>Concentration, mol L(^{-1})</th>
<th>Observed combustion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca(NO(_3))(_2)</td>
<td>5</td>
<td>Yes</td>
</tr>
<tr>
<td>KNO(_3)</td>
<td>3</td>
<td>Yes</td>
</tr>
<tr>
<td>Mg(NO(_3))(_2)</td>
<td>3</td>
<td>Yes</td>
</tr>
<tr>
<td>NaNO(_3)</td>
<td>7</td>
<td>Yes</td>
</tr>
</tbody>
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Combustion performed using 6 mL of H\(_2\)O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.
Table 3. Evaluation of combustion of filter paper (without sample) with different concentrations of nitrate solutions (50 µL) (n = 3).

<table>
<thead>
<tr>
<th>Igniter solution</th>
<th>Concentration, mol L$^{-1}$</th>
<th>Observed combustion</th>
<th>Ignition time, s</th>
<th>Combustion time, s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca(NO$_3$)$_2$</td>
<td>1</td>
<td>Yes</td>
<td>8.0 ± 1.0</td>
<td>7.9 ± 1.0</td>
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<tr>
<td></td>
<td>3</td>
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<td>5.3 ± 1.2</td>
<td>7.7 ± 1.5</td>
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<td></td>
<td>5</td>
<td>Yes</td>
<td>4.7 ± 0.6</td>
<td>7.0 ± 1.0</td>
</tr>
<tr>
<td>KNO$_3$</td>
<td>1</td>
<td>Yes</td>
<td>8.7 ± 1.2</td>
<td>7.0 ± 1.0</td>
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<tr>
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<td>3</td>
<td>Yes</td>
<td>8.0 ± 1.0</td>
<td>5.7 ± 1.5</td>
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<tr>
<td>Mg(NO$_3$)$_2$</td>
<td>1</td>
<td>Yes</td>
<td>6.0 ± 2.0</td>
<td>6.7 ± 2.1</td>
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<td>5.3 ± 1.5</td>
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<td>NaNO$_3$</td>
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<td>Yes</td>
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<td>3</td>
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<td>6.0 ± 3.0</td>
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<td>7</td>
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<td>6.3 ± 0.6</td>
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<tr>
<td>NH$_4$NO$_3$</td>
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<td>Yes</td>
<td>7.3 ± 1.5</td>
<td>6.0 ± 1.7</td>
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<td>9</td>
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<td>6.3 ± 0.6</td>
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<td></td>
<td>12</td>
<td>Yes</td>
<td>4.3 ± 0.6</td>
<td>6.0 ± 1.0</td>
</tr>
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</table>

Combustion performed using 6 mL of H$_2$O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.
Table 4. Evaluation of the effect of microwave power on the combustion of filter paper using 7 mol L\(^{-1}\) NH\(_4\)NO\(_3\) as igniter.

<table>
<thead>
<tr>
<th>Microwave Power, W</th>
<th>Observed combustion (replicates)</th>
<th>Ignition time, s</th>
<th>Combustion time, s</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Experiment 1</td>
<td>Experiment 2</td>
<td>Experiment 3</td>
</tr>
<tr>
<td>250</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>500</td>
<td>Yes</td>
<td>Yes</td>
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<td>750</td>
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<td>Yes</td>
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<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
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</table>

Combustion performed using 6 mL of H\(_2\)O as absorbing solution; microwave irradiation up to 60 s; 20 bar of oxygen pressure.
Table 5. Evaluation of different nitrate solutions for combustion of whole milk powder (100 mg) (n = 3).

<table>
<thead>
<tr>
<th>Igniter solution</th>
<th>Concentration, mol L^{-1}</th>
<th>Observed combustion</th>
<th>Ignition time, s</th>
<th>Combustion time, s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca(NO$_3$)$_2$</td>
<td>5</td>
<td>Yes</td>
<td>6.5 ± 0.5</td>
<td>5.5 ± 0.5</td>
</tr>
<tr>
<td>KNO$_3$</td>
<td>3</td>
<td>Yes</td>
<td>5.5 ± 0.5</td>
<td>7.5 ± 0.5</td>
</tr>
<tr>
<td>Mg(NO$_3$)$_2$</td>
<td>3</td>
<td>Yes</td>
<td>6.5 ± 0.5</td>
<td>6.5 ± 0.5</td>
</tr>
<tr>
<td>NaNO$_3$</td>
<td>7</td>
<td>Yes</td>
<td>5.0 ± 1.0</td>
<td>6.0 ± 1.0</td>
</tr>
<tr>
<td>NH$_4$NO$_3$</td>
<td>12</td>
<td>Yes</td>
<td>6.7 ± 0.6</td>
<td>7.5 ± 0.5</td>
</tr>
</tbody>
</table>

Combustion performed using 6 mL of H$_2$O as absorbing solution; microwave power of 1400 W up to 60 s; 20 bar of oxygen pressure.
Figure 1

- Gas release valve
- Quartz holder
- Sample + filter paper
- Absorbing solution

Window 13 cm
4 cm
Figure 2
Figure 3
Figure 4
Figure 5
IGNITER

Ca(NO₃)₂  NH₄NO₃
Mg(NO₃)₂  KNO₃  NaNO₃

COMBUSTION

MW