Faster, Cleaner, Safer: Why more labs are turning to microwave furnaces

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Conventional ovens and muffle furnaces are used daily in thousands of laboratories for many different applications from drying to ashing and are designed to provide continuous heating often for long periods. Microwave furnace systems can be used for exactly the same applications, albeit with significant advantages over conventional systems. Simon Osborne, from Analytix, examines the use of microwave furnace systems, the advantages they can offer over traditional systems, and why laboratories should consider these as an alternative when replacing their current technology.

To emphasise the benefits of microwave technology, we will examine one application that employs high temperatures, the determination of ash content. Whether for process control or as a preparation method for other analytical techniques, this is a relatively simple process. Many users however often comment on certain negative aspects of using conventional ovens or muffle furnace systems for this application, these being:

- Long ashing times creating a potential bottleneck in sample preparation
- Poor programmable temperature control and occasional incomplete ashing
- Safety problems with fumes from samples escaping into laboratory
- Heat from system affecting laboratory working conditions
- High energy consumption with systems left on continuously

With increasing constraints – time and financial – on laboratories and testing facilities, these perceived drawbacks that hamper operating time, cost, and working conditions, can become seriously detrimental to overall performance and turnaround rate of samples and test subjects.

Microwave-enhanced sample preparation fundamentals

A special microwave-transparent ceramic muffle sits inside the microwave cavity that allows microwave energy to pass through so the sample can be heated directly. In the top or side sections of the muffle sits a silicon carbide plate that efficiently absorbs microwaves and rapidly heats up. Porous ceramic honeycomb frits built into the side walls of the muffle furnace allow a constant stream of air to pass over the sample crucibles. The combination of direct microwave heating, the radiated heat from the silicon carbide plates, and the "superheating" from the oxygen in the air flow through the frits, results in a dramatic reduction of ashing times.

A high-precision thermocouple located in close proximity to the bottom of the crucibles (and unaffected by the airflow) monitors the temperature inside the furnace. The thermocouple is used as a feedback control mechanism to regulate microwave power output and maintain the user selected ramping/ashing temperature. The high-efficiency

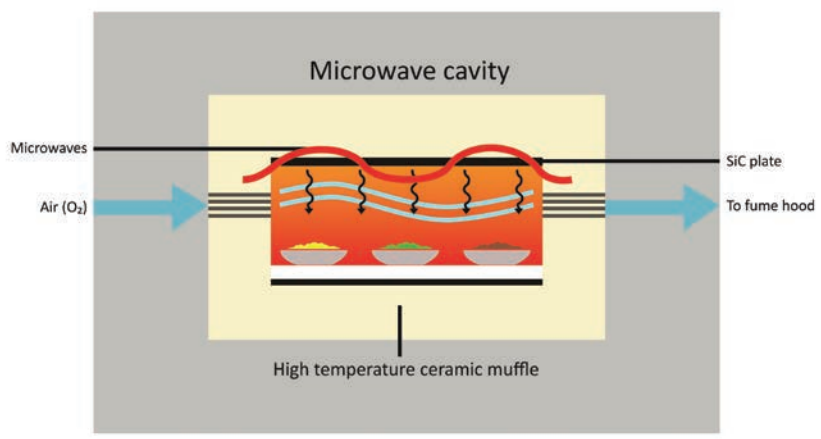

Figure 1: Modes of heating in a microwave furnace

microwave energy coupling characteristics of the silicon carbide plate allows the use of any type of crucible (metal, porcelain, disposable quartz fibre) within the microwave furnace.

Ashing times

Conventional ovens and muffle furnaces used in a huge number of laboratories for ashing, are inefficient in a number of respects that often leads to the process becoming tedious and time consuming. Users are often unaware how long the ashing process takes as samples are frequently left ashing overnight as the process cannot be completed during the working day.

With microwave ashing instruments, ashing can be completed many times faster than traditional techniques have previously allowed and in some cases the process can achieve a 97% reduction in ashing times that in turn will dramatically improve upon existing workflows. By reducing ashing times by such a margin, laboratories can dramatically reduce turnaround time on incoming samples, for example, giving much faster feedback to production processes.

Temperatures of 800°C can be reached within 30 minutes, dramatically reducing ashing times, often from hours to minutes, and offers the laboratory the most versatile way to ash samples.

Sample	Ashing Temperature (°C)	Microwave Ashing Time (minutes)	Traditional Ashing Time (minutes)
Pet food (ground)	575	21	90
Cat food	575	15	90
Polypropylene	650	22	80
PVC	900	15	120
Polyester	600	15	480
Polyurethane	900	15	120
Coal standard AR 2782	750	20	120
Dried egg yolk	925	25	240
Activated coal	750	25	180
Flour	900	50	360
Salami	600	60	360

Figure 2: Typical ashing time improvements with microwave furnace

The knock-on effect of this is a vastly increased capacity for sample throughput. High capacity muffle furnaces up to 5L are available so the ability to process up to fifteen 25ml crucibles simultaneously up to temperatures of 1200°C offers a faster, more efficient way of working and reduces 'waiting' time.

Temperature control

Conventional ovens and muffle furnace systems offer very good thermal stability and will maintain a constant temperature for extended periods. There is the possibility however for thermal gradients across an oven especially when filled with crucibles as they are heated from the outside surface and rely on a fan for temperature distribution. This can sometimes lead to incomplete ashing unless the ashing time is extended or the temperature is raised. As a worst case there might be the requirement to reprocess the samples. Some microwave systems are also heated from the side of the muffle by silicon

carbide plates whereas others are heated from the top that will then provide uniform heating across all samples with a lower temperature variability across the muffle.

Management of fumes

Ashing can produce large quantities of noxious and potentially toxic fumes and every precaution to reduce or eliminate exposure to these should be taken. Laboratories and testing facilities have long since led the way in terms of health and safety development, and microwave ashing represents another step forward in terms of safety and environmental comfort for analysts. A built-in exhaust system removes fumes and directs them through an exhaust hose to a fume hood or extraction system.

Sulphate ashing

Sulphate ashing introduces a potential additional health and safety hazard as sulphuric acid is used as a preliminary charring step and the removal of toxic acid fumes has to be considered. Microwave ashing systems are available with additional acid scrubbing modules specially engineered for this application. All steps of the process are automated that eliminates the need for the preliminary manual charring step to fume off sulphuric acid. The analyst weighs out the sample as normal into the crucible, adds 1ml sulphuric acid, loads the crucible into the microwave furnace and starts the pre-stored program. This eliminates the analyst exposure to acid fumes and minimises equipment corrosion and acid fume pollution. Whilst providing a safer working environment, ashing times can be reduced from 8-12 hours to 60-90 minutes.

Sample	Ashing Temperature (°C)	Microwave Ashing Time (minutes)	Traditional Ashing Time (minutes)
Lactose	600	60	480
NTF	800	50	480
Cellulose	800	80	500
Antibiotics	850	65	480
Tartaric acid	800	80	500

Figure 3: Typical sulphate ashing time improvements with microwave furnace

Some commercially available systems provide printed documentation and also the recall of the entire ashing process for quality control requirements. By removing the exposure to acid fumes, these systems can offer analyst the fastest, cleanest, safest, and most versatile way to ash a wide variety of samples.

Large sample amounts

The ashing of large amounts of samples such as polymers, petroleum, feeds, and pharmaceuticals presents a further challenge as these matrices generate a large quantity of fumes. In addition they often require an initial heating stage with a hotplate or Bunsen burner to reduce the volume and remove potentially noxious by-products. In an oven, the fumes condense when they come in contact with a cooler surface such as an extract fan, and will result in hard layers of material collecting. This will reduce the reliability and lifetime of the system and increase overall cost of running the laboratory. Microwave ashing systems are now available that use novel extraction methods where the materials that could cause potential problems are removed before they can damage cooler parts of the instrument.

Sample	Sample Amount	Number of samples	Reference Method	Ashing Temperature (°C)
Fuel Oil	Up to 25 grams	Up to 3	ASTM D482 - 95	775
Crude Oil	Up to 15 grams	Up to 3	ASTM D482 - 95	775
Polyethylene terephthalate	Up to 35 grams	Up to 3	ASTM D5630 - 94	900
Polyethylene	Up to 35 grams	Up to 3	ASTM D5630 - 94	900
Polypropylene	Up to 40 grams	Up to 3	ASTM D5630 - 94	900
Wheat flour	Up to 15 grams	Up to 3	ISO 2171	900

Figure 4: Ashing of large sample amounts

Environmental considerations - Heat

Traditional ovens have a tendency to diffuse heat while in use that can lead to an uncomfortable working environment. With a microwave furnace system, the microwave-transparent ceramic muffle furnace is an outstanding insulator and minimises heat loss and subsequent transfer to the surrounding area. Any heat generated on the outside of the muffle is removed from the system by the same method as fume extraction as described above. As an example, even with a temperature of 1000°C in the microwave muffle furnace, the outer chassis will be lower than 40°C and the ambient temperature of a laboratory will be unaffected, even when the unit is running at its highest temperatures.

Environmental considerations - Energy consumption

One consideration when investing in any new piece of equipment will always be the cost of ownership and that includes the running costs. The potential reduction of these associated with microwave ashing instruments is notable. Conventional ovens are often heating 24 hours a day (up to 5kW), because of the vastly increased speed and efficiency associated with microwave systems, they are able to operate for much less time. A typical microwave system's total power consumption over 6 hours at 700°C is under 1.3kW. That's before you even consider the speed improvements that themselves deliver operational and process cost savings.

Conclusions

Industry benchmarks change and improve all the time and now microwave technology is being implemented in a way that offers several significant advantages to sample preparation in the laboratory as it provides an excellent alternative to both conventional oven and muffle furnaces.

There is certainly a compelling case for making the switch to microwave furnace instruments as they can additionally offer a range of complementary improvements such as printing and storing the actual ashing profile for later retrieval. As described above, these improvements do not simply relate to speed, though that improvement is considerable; there are also gains to be made in terms of safety, environmental comfort, operating procedures, efficiency, throughput, and quality control reproducibility.

Analytix is the UK distributor of the Milestone Pyro, PyroSA and PryoXL microwave ashing systems. For more information contact Analytix on 0844 800 4220.

Benefits of ashing with Microwave Ashing Systems

- *Reduces ashing times by up to 97%*
- *Reduces operator exposure to fumes and heat*
- *Reduces energy consumption and subsequent costs*
- *Automated sulphated ashing*
- *Process a variety of different samples using any crucible type*

How a typical microwave ashing instrument works

A special microwave-transparent ceramic muffle sits inside the microwave cavity that allows microwave energy to pass through so the sample can be heated directly. In the top or side sections of the muffle sits a silicon carbide plate that efficiently absorbs microwaves and rapidly heats up. Porous ceramic honeycomb frits built into the side walls of the muffle furnace allow a constant stream of air to pass over the sample crucibles. The combination of direct microwave heating, the radiated heat from the silicon carbide plates, and the "superheating" from the oxygen in the air flow through the frits, results in a dramatic reduction of ashing times.

A high-precision thermocouple located in close proximity to the bottom of the crucibles (and unaffected by the airflow) monitors the temperature inside the furnace. The thermocouple is used as a feedback control mechanism to regulate microwave power output and maintain the user selected ramping/ashing temperature. The high-efficiency microwave energy coupling characteristics of the silicon carbide plate allows the use of any type of crucible (metal, porcelain, disposable quartz fibre) within the microwave furnace.

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