Rapid Analysis of Plastic Pellets

Abstract

Plastic is used in countless production processes, from extrusion and injection molding to blow molding and thermoforming. Although the processes vary widely, the need for process control and quality control is universal. Moisture content in raw and compounded plastic pellets has a direct impact on nearly all molding processes and must be tightly controlled. Purity levels in neat plastic are an indication of successful polymerization and can provide valuable information on catalyst consumption during polymerization. Master batch additives such as fillers, colorants, antioxidants, and plasticizers must be within specification for performance of the final product to be satisfactory. Filler content is typically quantified by ash analysis, whereas other additives such as plasticizers and antioxidants must typically be extracted from the polymer sample for analysis by chromatography.

This study demonstrates that CEM's rapid analysis technologies can analyze a wide range of plastics with reference-level precision and accuracy.

Technologies

SMART Q

The SMART Q™ quartz-halogen moisture analyzer (*1)* is uniquely designed to accurately measure low moisture levels, common in plastic pellets. With a highly accurate 4-place analytical balance and 3-digit moisture readout, the SMART Q provides reliable, repeatable results in approximately five minutes. The SMART Q uses direct sample temperature feedback and active cavity ventilation to dry samples faster than any other infrared moisture analyzer, with no cavity pre-heat.

To demonstrate the performance of the SMART Q, five types of plastic pellet samples were analyzed: nylon, ABS, polycarbonate, polypropylene and a rubber thermoplastic elastomer. The ABS, polypropylene and rubber TPE were compounded pellets filled with carbon black, whereas the nylon and polycarbonate were neat pellets. All pellets were subjected to ambient laboratory temperature and humidity for 24 hours, prior to analysis. For moisture determination, a 15 g sample of each product was analyzed in the SMART Q. Reference testing was performed in an air-oven in triplicate to establish a basis of comparison. The air-oven method was set for 8 hours at 100 °C, followed by a cooling period under desiccation to ensure complete drying.

Results for average percent moisture using the SMART Q compared closely to air-oven results, as illustrated in **Table 1** (page 2). The average absolute difference between the SMART Q results and air-oven results are less than 0.003%. The SMART Q precision outperformed the air-oven reference method, exhibiting average standard deviations of 0.011% and 0.013% respectively.

Table 1. Accuracy of SMART Q for Moisture Analysis of Plastic Pellets

Sample	Reference Moisture (%)	Reference STDEV	SMART Q Moisture (%)	SMART Q STDEV	Difference
Nylon (Unfilled)	1.090	0.015	1.087	0.015	-0.003
ABS (Black Filled)	0.323	0.022	0.328	0.010	0.005
Polycarbonate (Unfilled)	0.173	0.008	0.175	0.013	0.002
Polypropylene (Black Filled)	0.094	0.009	0.096	0.006	0.002
Rubber TPE (Black Filled)	0.062	0.010	0.061	0.012	-0.001

Phoenix

Using microwave technology to quickly and efficiently generate heat, the Phoenix™ microwave muffle furnace (*2*) can reach optimal ashing temperature in less than 30 minutes, and ash samples in 10 minutes, when they typically take 1–2 hours in traditional muffle furnaces. With programmable methods that can set ramps and dwell times, the only steps the user has to take is weighing the sample and placing it in the muffle furnace. The Phoenix will ash the sample and calculate the results, ensuring the right result every time.

In a study comparing the time savings of the Phoenix microwave muffle furnace with traditional muffle furnaces, 8 polymer and compounding samples were ashed until completion, with the total time in furnace shown below in **Table 2**. The Phoenix provides significant time savings, with some samples being up to 97% faster.

Table 2. Phoenix Typical Ashing Times

Material	Conventional (minutes)	Phoenix (minutes)	Time Savings (%)
Butyl Rubber	90	20	78
Carbon Black	960	90	91
Polyester (Filled)	480	15	97
Polyethylene (Unfilled)	30	5	83
Polyethylene (% Carbon Black)	30	7	77
Polypropylene	30	5	83
Silicon Carbide Mixture	120	10	92
Stearates	90	5	94

EDGE

The EDGE® automated solvent extraction system (*3*) is the most advanced extraction system available. By combining pressurized fluid extraction and dispersive solid phase extraction, the EDGE is able to drastically reduce the sample preparation time and potential for human error. The result is fast, simple, and efficient extractions. In just 15 minutes, including filtration, cooling and system washing, additives such as plasticizers, antioxidants, and slip agents can be extracted from a wide variety of plastic types for rapid quality assurance.

To demonstrate the performance of the EDGE, a selection of polyvinyl chloride (PVC) certified reference materials (CRM) containing various phthalates was purchased and extracted for GCMS analysis. For each sample, a S1 Q-Disc® stack (C9+G1+C9 sandwich) was placed into the Q-Cup® base, and the two parts were screwed together, creating a seal between the Q-Cup and Q-Disc. One gram of CRM was weighed into an assembled Q-Cup. The Q-Cups were placed in the EDGE removable rack, each with a collection vial, and the rack was slid into place on the EDGE and queued for extraction.

The percent recovery of the extraction of phthalates from polyvinyl chloride is shown below in **Table 3**. All methods for sample prep, extraction, and analysis were based on CPSC-CH-C1001-09.1. For the extraction process, the total method time, including extraction, filtration, and system washing, never exceeded 15 minutes. The total extraction volume was 30 mL, and additional 20 mL of solvent was used to wash the system.

Table 3. Percent Recovery Data for Polyvinyl Chloride

Standard Compound	Average Recovery (n=6)	RSD
Dimethyl Phthalate	86	1.9
Diethyl Phthalate	83	1.5
Bis (2-ethylhexyl) Phthalate	88	1.5
Butylbenzyl Phthalate	86	1.7
Di-*n*-butyl Phthalate	84	2.5
Di-*n*-octyl Phthalate	101	1.7

MARS 6

The MARS 6™ microwave digestion system (*4*) provides the high temperatures needed to fully digest all polymer types. From nylon to PET, the system will provide a clear and particle-free liquid using only nitric acid. Traditional methods typically call for sulfuric acid, which is viscous and difficult to aspirate into ICP and ICP-MS systems. Some polymers such as polypropylene can be digested in this high-capacity vessels system, which can digest up to 40 samples simultaneously. Microwave digestion is complete in 30–45 minutes for most polymer samples, which are then ready for dilution and analysis.

PET is a member of the polyester family. The presence of a large aromatic ring and repeating units give it significant strength and stability, but makes it very difficult to digest. It has numerous applications from plastic soda bottles and food containers, to carpet and furniture. The use of heavy metals, particularly Antimony (Sb) in the synthesis of PET, require metals testing, as it relates to public health.

A total of four PET samples were digested in a single batch in a MARS 6 microwave system using iWave® contactless, *in-situ* temperature measurement. iWave is a novel technology advancement that utilizes Light Emitting Technology™ to measure the temperature of the actual sample solution inside the vessel, and does not require an internal probe.

Approximately 0.25 g of four PET samples were weighed into an iPrep® vessel liner and 10 mL of concentrated nitric acid was added to each. The vessels were capped and assembled and then placed in the MARS 6 for digestion.

Figure 1 illustrates that all samples were completely digested, producing clear, colorless solutions that are ready for further analysis.

Conclusion

For plastics applications where accuracy and precision are critical, CEM offers reliable solutions that match longer reference methods in only a few minutes. CEM's combination of proprietary and patented technology translates into one of the fastest, easiest to use products on the market.

PET Pellets
Max Size: 0.25 g
Result: Clear

Before

After (sample diluted to 50 mL)

Figure 1. PET Pellets Before and After Digestion

References

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