

The Extraction of Antioxidants from Food Packaging Film

Abstract

Polymer materials are an integral part of every day life, especially food packaging. Often, antioxidants are added to food packaging materials to help aid in their stability. However, these antioxidants can leach out of the materials into food, demonstrating the importance of being aware of the levels of antioxidants in the polymer materials in food packaging. In this work, Irganox 1076 and Irganox 1010 were extracted from food film provided by a local manufacturer, using three CEM extraction systems, the MARS 6™ microwave system, the Discover Prep™ microwave system, and the EDGE® automated extraction system. The MARS 6 method has been long proven in the industry and is known to deliver accurate results. The results obtained for the extractions completed by Discover Prep and the EDGE were compared to the MARS 6 results, and it was found that the Discover Prep and the EDGE extracted the additives from food film with comparable recoveries and low standard deviations. The MARS 6, the Discover Prep, and the EDGE are each excellent options for the extraction of antioxidants from polymers.

Introduction

Polymer materials are commonly found in our day-to-day products. Consumers interact with them quite often, from car parts, to children's toys, to food packaging. Many foods purchased in the grocery store are wrapped in some sort of plastic material. For food packaging, additives, such as antioxidants, are often added to help maintain their integrity and stability. However, with exposure to heat and over time, these additives can leach out of the materials and into food. Thus, accurate methods for assessing levels of antioxidants in food packaging is important.

In this work, three CEM products, the MARS 6 microwave system, the Discover Prep microwave system, and the EDGE automated extraction system, were used to extract two antioxidants, Irganox 1076 and Irganox 1010, from food film packaging provided by a local manufacturer. The MARS 6 extraction methodology has long been known as a valid extraction technique, starting with soils almost 30 years ago. Extraction applications have grown to many other areas, including plastics, and it is regarded as a method producing 100% recovery. The MARS 6 and the Discover Prep incorporate microwaves into their extraction, while the EDGE utilizes a pressurized system. The results from the Discover Prep and EDGE extractions were compared to the MARS 6 results, and it was found that the other two CEM instrument choices produced similar results with low standard deviations. Thus, CEM provides three excellent choices for the extraction of antioxidants from polymers.

Materials and Methods

Reagents

Food film was provided by a local manufacturer. ASTM D6042-96 Calibration Mix was purchased from Restek. ACS-grade 2-propanol and HPLC-grade 2-propanol were purchased from Sigma. 5-mL syringes were purchased from Fisher, and 0.2-micron PTFE Luer lock syringe filters were purchased from Phenomenex. Other materials were provided by CEM.

MARS Extraction

A portion of 1 g of food film, cut into 0.5-cm wide squares, was weighed directly into a 110 mL MARSXpress™ Plus vessel, and 20 mL of ACS-grade 2-propanol were added. Samples were prepared in quadruplicate. Each vessel was loaded into a MARSXpress™ Plus turntable. The samples were extracted using a OneTouch™ method with the following parameters: temperature of 120 °C, sample type of organic, and hold time of 59 minutes, 59 seconds. After extraction, the extracts were decanted into a 30 mL syringe prepared with a 0.2-micron PTFE Luer lock syringe filter and were filtered into evaporation vials. 1 mL was removed for the analysis of Irganox 1076. The remaining volume of each extract was evaporated, using a nitrogen evaporator set at 50 °C, using a flow rate of 2.5 mL/min. Once the extracts were below 1 mL, they were transferred to 2 mL volumetric flasks and brought up to volume, using HPLC-grade 2-propanol. The dried extracts were transferred to amber vials for the analysis of Irganox 1010.

Discover Prep Extraction

A portion of 1 g of food film, cut into 0.5 cm-wide squares was weighed directly into a 35 mL Discover Pyrex vial, and 20 mL of ACS-grade 2-propanol were added. Samples were prepared in quadruplicate. Each sample was loaded into a Discover Prep Autosampler rack and extracted using the following Discover Prep Method: standard method control type, temperature of 120 °C, time of 30 minutes, and cooling temperature of 55 °C. After extraction, the extracts were decanted into a 30 mL syringe, prepared with a 0.2-micron PTFE Luer lock syringe filter and were filtered into evaporation vials. A portion of 1 mL was removed for the analysis of Irganox 1076. The remaining volume of each extract was evaporated, using a nitrogen evaporator set at 50 °C, using a flow rate of 2.5 mL/min. Once the extracts were below 1 mL, they were transferred to 2 mL volumetric flasks and brought up to volume, using HPLC-grade 2-propanol. The dried down extracts were transferred to amber vials for the analysis of Irganox 1010.

EDGE Extraction

A portion of 1 g of food film, cut into 0.5 cm-wide squares was weighed directly into a Q-Cup®, prepared with the S1 stack of Q-Discs®. A Q-Screen® was applied above each sample. Samples were prepared in quadruplicate. Each Q-Cup was loaded into an EDGE rack, along with an amber extraction vial. The samples were extracted, using the EDGE method below. After the extraction, 1 mL was removed for the analysis of Irganox 1076. A portion of 15 mL of the remaining extract was removed and transferred to an evaporation vial. The extracts were evaporated, using a nitrogen evaporator set at 50 °C, using a flow rate of 2.5 mL/min. Once the extracts were below 1 mL, they were transferred to 2 mL volumetric flasks and brought up to volume, using HPLC-grade 2-propanol. The extracts were then transferred into 5 mL syringes, prepared with a 0.2-micron PTFE Luer lock syringe filter and were filtered into amber vials for the analysis of Irganox 1010.

EDGE Method for Antioxidants from Polymer Film

Cycle 1

Extraction Solvent: 2-Propanol

Top Add: 20 mL

Bottom Add: 0 mL

Rinse: 0 mL

Temperature: 120 °C

Hold Time: 60:00 (mm:ss)

Wash 1

Wash Solvent: 2-Propanol

Wash Volume: 10 mL

Temperature: 120 °C

Hold Time: 00:30 (mm:ss)

Wash 2

Wash Solvent: 2-Propanol

Wash Volume: 10 mL

Temperature: - - -

Analysis

For analysis, a Waters ACQUITY UPLC H-Class System with a PDA Detector was used. A Restek Ultra C8 5 µm (150 x 4.6 mm) column was utilized for the separation of Irganox 1076 and Irganox 1010. The injection volume was 10 µL. The analytes were monitored at 200 nm, and the separation method using water as mobile phase A and acetonitrile as mobile phase B is indicated in **Table 1**. To assess the concentration of Irganox 1076 and Irganox 1010, a calibration curve in 2-propanol was created, using the Restek ASTM D6042-96 Calibration Mix from concentrations 7.5 µg/mL to 20 µg/mL. For each extraction type, a direct analysis was done to assess the concentration of Irganox 1076, and the concentration of Irganox 1010 was assessed from the concentrated extracts. An example chromatogram for the analytes is shown in **Figure 1** (page 3).

Table 1. Waters ACQUITY UPLC H-Class System Separation Program

Time (min)	Flow (mL/min)	%A
Initial	0.5	40
06:00	0.5	0
40:00	0.5	0
40:01	0.5	40
44:00	0.5	40

Results & Discussion

Using the MARS 6 method as the baseline for 100% recovery, the results from the Discover and EDGE extractions were compared to the results determined for the MARS 6. The recovery results are presented in **Table 2** for both the Discover Prep and the EDGE. The results obtained for both Irganox 1076 and Irganox 1010 indicated that both the Discover Prep and EDGE were able to extract the antioxidant compounds with extraction efficiencies similar to those found for the MARS 6 method. The results were also found to have relative standard deviation values less than 10% for all recovery values; thus, the recovery values were precise.

Table 2. Recovery Results for the Discover Prep and EDGE

Extraction Instrumentation	Analyte	Average Recovery	Relative STDEV	n
Discover Prep	Irganox 1076	86.83%	2.29%	4
Discover Prep	Irganox 1010	97.71%	4.83%	4
EDGE	Irganox 1076	74.94%	3.57%	4
EDGE	Irganox 1010	121.19%	8.68%	4

Conclusion

The analysis of antioxidants in food packaging is critical, because antioxidants can leach from food packaging. In this work, the MARS 6 microwave system, the Discover Prep microwave system, and the EDGE automated extraction system were all used to extract the antioxidants Irganox 1076 and Irganox 1010 from food film, provided by a local manufacturer. The results provided by the MARS 6, were regarded as the “true” value for each antioxidant. The values obtained for the extractions done by the Discover Prep and EDGE systems were compared to the MARS 6 values, and it was found that the recoveries for these systems were all greater than 70%, indicating a good extraction. The relative standard deviations were also less than 10%, indicating a repeatable extraction. Thus, CEM has three excellent options for the extraction of antioxidants from food packaging film.

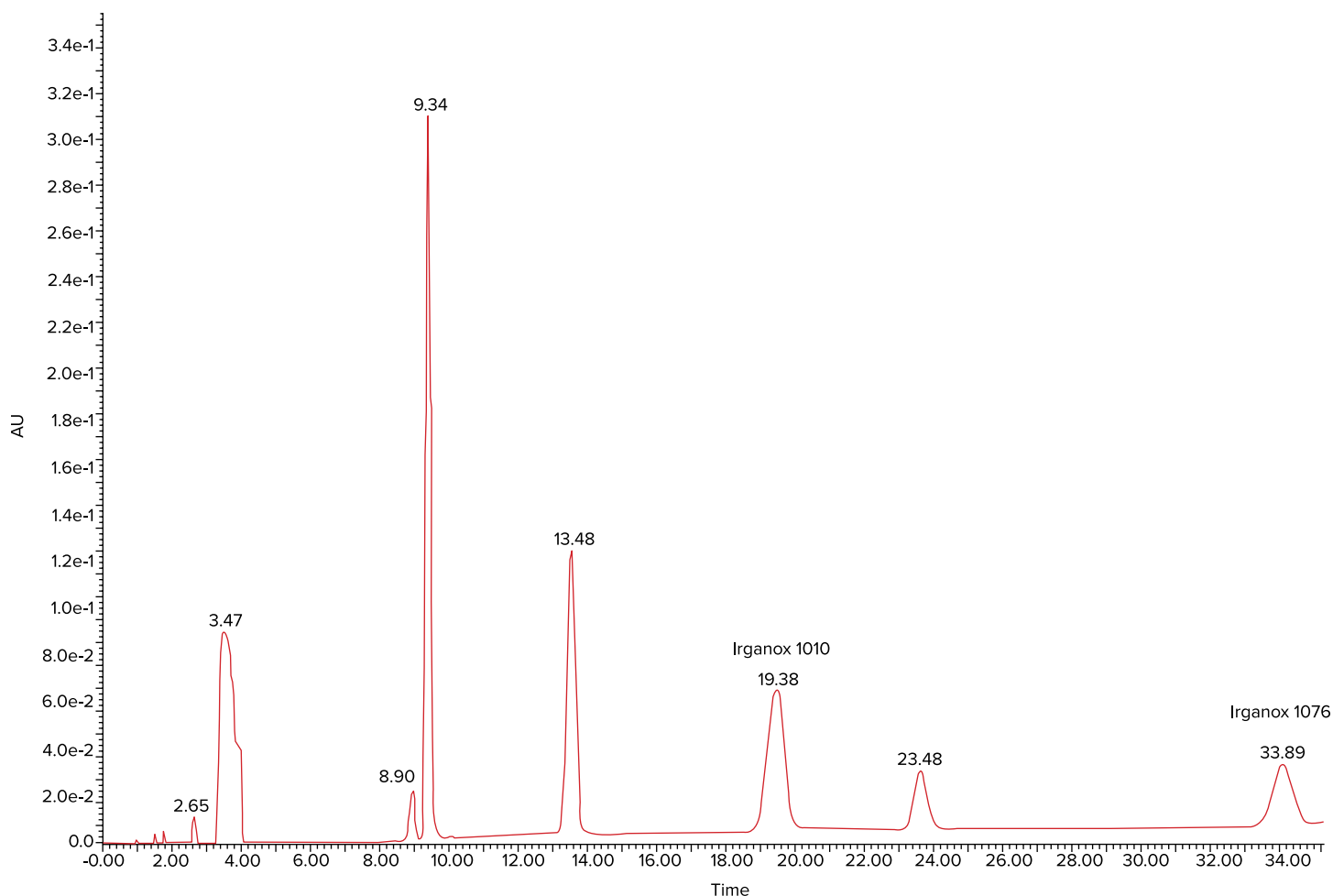


Figure 1. Example Chromatogram of Restek ASTM D6042-96 Calibration Mix

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