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Enhancing natural product extraction and mass transfer using selective microwave heating

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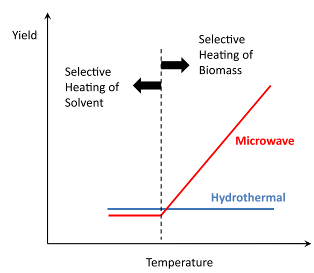
HIGHLIGHTS

- Conventional and microwave extraction are comparable with no selective heating of biomass.
- A step-change in yield is observed with microwave heating at temperatures that ensure selective heating.
- Dielectric property data correlates with the microwave extraction performance.
- Selective heating leads to a reduction in water chemical potential within biomass.
- Equilibrium cell pressures exceeding 100 bar can be achieved with a temperature difference of 1 °C.

GRAPHICAL ABSTRACT



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ABSTRACT

This study uses a combination of empirical observations and an analysis of mass transfer behaviour to yield new insights into the mechanism of microwave assisted extraction. Enhancements in extraction rate and yield were observed experimentally compared with conventional extraction at temperatures in excess of 50 °C, however at lower temperatures there was no observable difference between the two processes. A step-change in extract yield between microwave and conventional processes was shown to be caused by selective heating. A temperature gradient of the order of 1 °C is sufficient to reduce the water chemical potential within the cell structure, which changes the osmotic potential such that internal cell pressures can increase to the point where disruption occurs. This paper demonstrates the need to operate microwave extraction processes at a temperature that enables selective heating, and a newly-proposed mass transfer phenomenon that could have wider positive implications for extraction and leaching processes.

1. Introduction

There is an increasing interest in the use of extraction processes to produce natural compounds from plant materials as a sustainable alternative to direct chemical synthesis. Extraction is carried out using a wide range of solvents and processing methods, and a significant number of studies have focussed on the use of microwave assisted extraction (MAE) instead of conventional hydrothermal processes. Improvements in the rate of extraction and quality of the extract have been reported for applications in gas

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and oil, food and medicine and perfume and flavour industries when microwave heating was used (Flórez et al., 2015; Farhat et al., 2011). Reductions in energy requirements, solvent loading and by-product formation have also been well documented when MAE processes are used compared with hydrothermal or solvent based extraction systems (Rombaut et al., 2014). Microwave heating has also been shown to lead to wider process engineering benefits when carried out at scale, such as a reduction in equipment size and simplification of processing steps when compared to conventional methods, which results in further economic benefits (Robinson et al., 2009). It is widely accepted that the selective heating and resulting differences in the heat and mass transfer gradient, coupled with thermally induced structural damage of the matrix, play a role in the enhanced yield and reaction rates that MAE facilitates (Flórez et al., 2015). Cell-rupture is widely thought to occur when microwave heating is used (Osaili, 2012; Chemat and Cravotto, 2013; Mandal et al., 2015), however there has been limited consideration of how cell rupture can be induced when microwaves are applied, or that the extraction mechanism may vary depending on the material treated and the nature and location of the extract with the plant matrix. MAE can be used to extract lipids, polysaccharides, polyphenolics, protein and essential oils (Flórez et al., 2015). This paper considers the overall extraction yield from okra pods using neutral aqueous extraction. Okra pod hydrocolloid has been extracted using MAE at a yield of 14%, but extensive pretreatment (conventional heating, air drying, grinding and refluxing in 80% ethanol for 5 hours) was carried out and results were not compared with yields from conventional solvent extraction (Samavati, 2013). The main component of okra extracts are polysaccharides (Lee et al., 2015). MAE of polysaccharides such as pectin is of particular interest due to the inherent difficulty in extraction leading to the requirement for conventional extraction techniques to use extremes in pH, and high temperatures and pressures. Numerous workers have investigated the use of MAE to extract polysaccharides from plant materials. The yield of pectins from orange peel using MAE is reported to be up to 250% higher compared with that of conventional solvent extraction (Kratchanova et al., 2004). Several papers cite vapour formation in the capillary-porous structure resulting in large pressure build-up and swelling of the cells as the primary reason for the increase in extraction yield (Kratchanova et al., 2004; Zhongdong et al., 2006; Yeoh et al., 2008; Yan et al., 2010; Rodriguez-Jasso et al., 2011), and Scanning Electron Microscope images are often used to support this theory. However, it is often difficult on close examination to see any definite difference in the images obtained from microwave and conventionally treated samples. Fishman et al. Fishman et al. (2006) carried out microwave extraction of lime flavedo, albedo and pulp under pressure (up to 50 psi) and concluded that MAE occurs via conventional acid extraction, albeit accelerated by the unique heating profiles induced by microwave heating.

There have been extensive studies on the chemical potential and mass transfer of solvent/solute systems across membrane and cell structures (Wijmans and Baker, 1995; Elmoazzen et al., 2009; Welti-Chanes et al., 2002), however this approach has not previously been used to consider the temperature gradients that can exist when microwave heating is used. Dielectric properties, which quantify the interaction between microwave energy and process materials, are rarely measured yet are essential to understand the heating behaviour and thermal gradients that can exist within the system (Meredith, 1998). To date, there has been no attempt to determine quantitatively how microwave heating can lead to enhanced cell rupture, and this paper makes a significant contribution to the field by addressing this question.

The aim of this study is to investigate and understand the extraction mechanisms in solvent-based systems by comparing microwave and conventional extraction methods, analysing the empirical observations against the dielectric properties of the system components, and understanding the fundamental mass transfer properties of the system by analysing the effect of thermal gradients on chemical potential.

2. Material and methods

2.1. Materials

Okra, sourced from UK food importers, was used as a model feedstock for this study, and the yield of extractable compounds was used as an indicator of process performance and for comparison between microwave and conventional extraction methods. Before processing the okra was washed with deionised water, the upper crown head and the seeds were removed and the pods sliced into uniform sizes.

2.2. Extraction

Sliced okra and deionised water were loaded into a Pyrex extraction cell and sealed with a plastic lid. The cell was placed within a Miniflow 200SS (Sairem, France) microwave heating system operating at 2450 MHz. A temperature probe was inserted into the flask to monitor the bulk temperature of the solvent (water). Note that it was not possible to measure the temperature of the okra during treatment. The power applied to the system was varied to achieve a desired temperature set-point.

After the predetermined extraction time the flasks were kept aside at room temperature for 1 hour for cooling and complete release of the extractable solutes into water (Ameena et al., 2010). Aqueous extract was recovered from the processed mixture by centrifugation at 4000 rpm for 30 minutes. A sample of aqueous extract was dried at 105 °C until a constant solid mass was attained, and extract yield on dry basis (DB) was calculated based on the solid extract mass relative to the dry mass of feedstock. A sample of the feedstock was dried at 105 °C until a constant mass was obtained to calculate the moisture content in the fresh okra, which was 90.93%. Conventional hydrothermal extraction of aqueous extract from sliced okra conducted in a water bath shaker was reported in a previous study (Lee et al., 2015) and the extraction data was used for comparison with the MAE results.

2.3. Dielectric property measurement

Dielectric property measurements were carried out independently of the microwave extraction measurements. A resonant cavity perturbation method was used to measure the dielectric properties of okra from 20 to 100 °C. It consists of a cylindrical copper cavity connected to a vector network analyser, which measures the frequency shift and change in quality factor relative to the empty resonating cavity when a sample is introduced. Samples were loaded into a quartz tube, and held in a conventionally heated furnace above the cavity until the temperature set-point was reached. The tube was then moved into the cavity, and the properties determined at 2470 MHz, which is within 20 MHz of the microwave heating equipment used in this study. The dielectric properties of deionised water were measured using an Agilent 8753 ES Vector Network Analyser and Coaxial Probe.