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DIELECTRIC MONITORING OF MICROWAVE EXTRACTION PROCESSES

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ABSTRACT

Our research investigated the dielectric traceability of microwave and ultrasound intensified extraction processes of plant by-products. For the experiments, I used plant by-products from beetroot, carrot and raspberry, which were extracted in 5% suspensions. The dielectric behaviour of the extracts was investigated in the frequency range 300-2400 MHz using an open-ended coaxial probe. For both the ultrasonic and microwave intensified procedures, the reported energy was 30, 45 and 60 kJ, respectively. Based on my research results, I found that the dielectric constant measured in the frequency range 700-900 MHz is closely related to the yield of total polyphenol and pectin regardless of the feedstock and treatment used, but the method has limitations due to the presence of components that affect the physical structure and the concentration of target components below the limit of detection.

Keywords: microwave, extraction, dielectric properties, dielectric measurements

1. INTRODUCTION

A significant proportion of agricultural and food by-products and wastes of plant origin contain valuable components (bioactive substances, heteropolysaccharides, etc.) that can be recycled for industrial use through appropriate recovery, separation and purification technologies, thus fitting into the concept of circular economy [1]. One of the most commonly used methods for recovering the various components that can still be used is extraction, whereby the target components are introduced into the liquid phase extract using a solvent, usually a selective solvent, and under suitable reaction conditions [2]. In order to improve the efficiency of extraction, novel, complementary methods are nowadays being used to achieve higher product concentrations with significantly lower solvent requirements and operation times. Examples of such intensification operations are microwave-assisted or ultrasound-assisted extraction, where the thermal effects generated by the microwave field in the former and the mechanical shear forces and cavitation phenomena in the liquid phase caused by ultrasound in the latter result in the destruction of the structure of the material to be extracted, which facilitates the recovery of the components. In the extraction of plant by-products of agro-food origin, the concentration of the target components to be extracted can be many times higher than the maximum concentration achievable in conventional extraction processes due to the plant cell wall disintegration induced by these intensification operations [3].

At the same time, the complexity of the processes justifies the need for real-time monitoring and control of the extraction operations to detect possible errors and improve the efficiency of each extraction operation. Monitoring techniques based on the dielectric behaviour of materials are becoming more and more widely used because they are fast, accurate, non-sampling, non-destructive and have applications in food processing, among others [4]. The dielectric properties of materials are already used today for the optimisation of extraction processes (adjustment of solvent concentration, pH, extraction time, etc.) [5], but research on continuous monitoring of the process based on dielectric parameters is still scarce. Based on these considerations, the focus of my current research is to monitor the microwave and ultrasound intensified extraction processes of different by-products of plant origin based on the dielectric behaviour of the extracted extracts.

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2. MATERIALS AND METHODS

For the extraction processes, we used three different plant by-products as feedstock, taking into account sustainability aspects: peelings from peeling beetroot and carrots, and the residual pulp after pressing raspberries for pulping. The residues of root vegetables were chopped to a final particle size of 3-5 mm prior to extraction, and 5% aqueous suspensions were prepared from all the raw materials included in the study. The extraction processes were intensified by microwave treatment at 2.45 GHz (Labotron 500) at 2 different power levels (250 W and 500 W) and by ultrasonic extraction at 200 W (Hielscher UP200S, 24 kHz). In order to compare the monitoring of extraction operations based on different principles, the principle of energy equivalence was applied. The times of the treatments with the intensification operation were chosen so that the energy transferred to the material was 30, 45 and 60 kJ. Thus, we monitored the different extraction processes by reaching these energy levels.

Pectin and total polyphenol concentrations were determined in the obtained extracts on a dry matter basis. Pectin was determined by spectrophotometric method using m-hydroxydiphenyl reagent, total polyphenolic content (TPC) was determined using Folin-Ciocalteu reagent. In parallel, the interaction between the components in the aqueous phase and the electromagnetic field was investigated using a laboratory dielectric measurement system. Using an open-ended coaxial dielectric sensor (DAK 3.5, SPEAG GmBh) connected to a vector network analyser (ZVL-3 VNA, Rhode&Schwarz GmBh), we determined the dielectric constant (ε') values of the extracts in the frequency range between 300 MHz and 2400 MHz.

3. RESULTS AND DISCUSSION

Based on our previous research and the available literature, it can be concluded that in the frequency range of 300-2400 MHz the dielectric behaviour is mainly determined by ionic conduction and dipole rotation, however, considering that the general frequency-dependent trends of dielectric properties in multicomponent heterogeneous systems are mostly not verifiable, only certain discrete frequency bands can be reliably used for monitoring physical, chemical, biological processes. In the monitoring of extraction processes, the frequency-dependent spectrum of the dielectric constant of the extract showed a local maximum at 800 MHz, independent of the type of treatment, the amount of energy applied and the feedstock (Figures 1-3). It can be concluded that the target components under investigation, polyphenols and pectin, have a significant effect on the dielectric behaviour in this frequency range, and therefore I used the data measured in the 700-900 MHz frequency range to compare the dielectric changes.

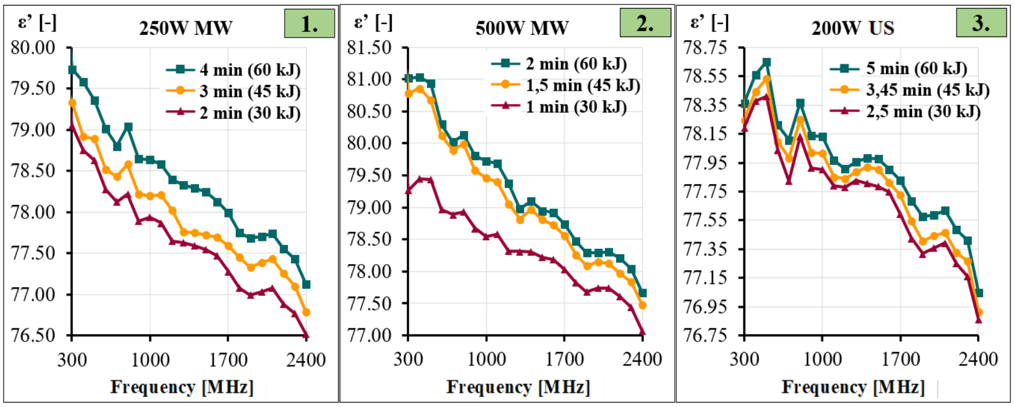

Figure 1-3. *Dielectric spectra of beet extracts obtained by different intensifying methods*

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With the progress of the microwave and ultrasound intensified extraction process of beetroot, the extraction indexes of the two tested components varied with an increasing trend, and we found that the polyphenol content of the extract exceeded the pectin concentration by 1.5-2 times during the whole extraction process. During the monitoring of the microwave extraction at 250 W power, there is a strong positive correlation between the variation of the dielectric constants measured at the inflection point of the dielectric spectrum (800 MHz) and its discrete frequency interval (700-900 MHz) (Figure 1) and the increase in the yield concentration of the tested components (Figure 4).

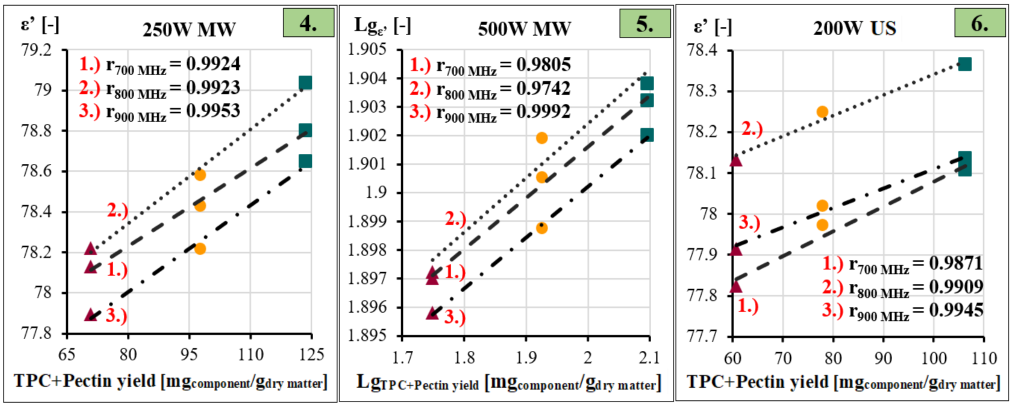

Figure 4-6. *Correlation analysis of beet extraction monitoring*

The increased (500 W) microwave power, presumably due to the different temperature rise tendencies, the relationship between the dielectric constant and the yield indices of the extract was approximated by a second-order polynomial fit. Therefore, for a proper regression and correlation analysis, the values were plotted using a logarithmic transformation, linearized, and then, based on the values of the point-fitted linear correlation coefficient, it was concluded that there was a close relationship between the variables (Figure 5). The frequency-dependent spectrum of the dielectric constant of the extract obtained after ultrasonic treatment followed a similar trend to the characteristic variation observed for microwave extraction, but due to the different disintegration effect, the local maximum value of ε' was recorded not only at 800 MHz but also at 500 MHz (Figure 3). The relationship between the dielectric constant values measured in the 700-900 MHz frequency interval and the variation of the yield indices during the ultrasonic extraction process also showed a close linear correlation (Figure 6).

In carrot extracts, the extraction of the tested components showed low absolute concentrations ($c_{max\ (pectin)} < 33 mg/g_{dm}$, $c_{max\ (polyphenol)} < 13 mg/g_{dm}$) and, similar to beet extraction, increased with increasing energy input, while the pectin content exceeded the polyphenol content. As the extraction progressed, there were no significant differences between the extraction indices and therefore no significant differences in the dielectric behaviour of the extracts obtained with different energy concentrations were observed at frequencies above 1100 MHz. The monitoring of microwave treatment at 250 W power and ultrasonic extraction showed that the correlation coefficient between the dielectric constant measured in the frequency range 700-900 MHz and the variation of the extraction indexes of the target components was high (r=0.95-0.99), i.e. the correlation is close and the relationship between the variables can be approximated linearly. For the 500 W microwave treatment, the relationship between the parameters varied according to a second-order function due to the different temperature rise profile.

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In all the raspberry pulp extractions studied, even at low energy concentrations, a significant amount of pectin was dissolved in the aqueous phase, in terms of dielectric properties, which probably masked the effect of other components (including polyphenols) on the dielectric behaviour. Furthermore, the pectin, due to its gelling effect, increased the viscosity of the raspberry extracts, thus changing the physical structure of the raw material to such an extent that the characteristic molecular mechanisms could not be validated in the frequency range I have investigated.

4. CONCLUSIONS

Based on the results of my research so far, I have concluded that dielectric parameters measured in the 700-900 MHz frequency band can be used to monitor the extraction process, regardless of the type of intensification operation. The variation of the dielectric constant showed a close correlation with the total polyphenol and pectin extraction indexes of beet and carrot extracts, thus concluding that the dielectric measurement method is potentially suitable for monitoring the extraction process. However, the specificities of the different raw materials of different origins included in the study have highlighted that the presence of components affecting the physical structure (e.g. pectin in the extraction of raspberries) and the difference in the concentrations of the extracts below the limit of detection may be limiting factors for the applicability of the method. This suggests that further experiments over a wider frequency range and other measurement set-ups are needed to clearly establish the relationship between the parameters under investigation and to develop reliable monitoring methods based on dielectric parameters, including for industrial practice.

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REFERENCES

[1] Ben-Othman, S., Jőudu, I., & Bhat, R. (2020). Bioactives from agri-food wastes: present insights and future challenges. *Molecules*, 25(3), 510.
[2] Jha, A. K., & Sit, N. (2022). extraction of bioactive compounds from plant materials using combination of various novel methods: a review Trends in Food Science & Technology, 119, 579-591.
[3] Singh, A., Nair, G. R., Liplap, P., Gariepy, Y., Orsat, V., & Raghavan, V. (2014). Effect of dielectric properties of a solvent-water mixture used in microwave-assisted extraction of antioxidants from potato peels. Antioxidants, 3(1), 99-113.
[4] Routray, W., & Orsat, V. (2018). recent advances in dielectric properties-measurements and importance. current opinion in food science, 23, 120-126.
[5] Álvarez, A., Fayos-Fernández, J., Monzó-Cabrera, J., Cocero, M. J., & Mato, R. B. (2017). Measurement and correlation of the dielectric properties of a grape pomace extraction media: effect of temperature and composition. Journal of Food Engineering, 197, 98-106.

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