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IDENTIFICATION OF CAPSAICIN FROM THE ALCOHOLIC AND OILY EXTRACTS OF HOT PEPPERS USING FTIR SPECTROSCOPY

BY

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Abstract. In this study, an attempt was made to identify capsaicin from the hot pepper alcoholic and oily extracts, using FTIR spectroscopy. Both the oily and alcoholic maceration were preserved for 5 years. The spectra obtained show characteristics bands that confirm the presence of functional groups specific to capsaicin. The antioxidant activity of the alcoholic hot pepper extract was also determined by the spectrophotometric method using 2,2-diphenyl-1-picrylhydrazyl (DPPH). Even after a long period of preservation, the alcoholic extract of hot pepper has a high antioxidant activity, which can be used to the preparation of cosmetic and food products to significantly increase their antioxidant properties and have a positive impact on the body.

Keywords: antioxidant activity over time, capsaicin, hot pepper, spectrophotometric analysis.

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1. Introduction

It is well known that hot pepper is a vegetable material rich in active compounds with specific properties. Thus, capsaicin (Fig. 1) is known as the alkaloid responsible for the spicy taste of hot peppers, its concentration varying depending on the plant breed, atmospheric and soil conditions. Capsaicin is also known for its remarkable medicinal properties, due to its high antioxidant activity, this being the reason for which it was considered a special constituent for traditional Chinese medicine (Taira *et al*., 2012), and for India to intensify the production of hot peppers, up until today being one of the largest producers in the world (Maurya *et al*., 2014; Yatung *et al*., 2014; Dudwal 2021).

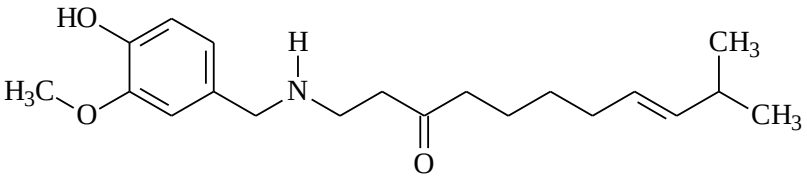

Fig. 1 – Chemical structure of capsaicin (Cernatescu and Cobzaru, 2014).

The antioxidant activity can also be provided by other compounds in the composition of hot peppers such as: ascorbic acid, tocopherol, β-carotene (Fig. 2), flavonoids and phenolic acids.

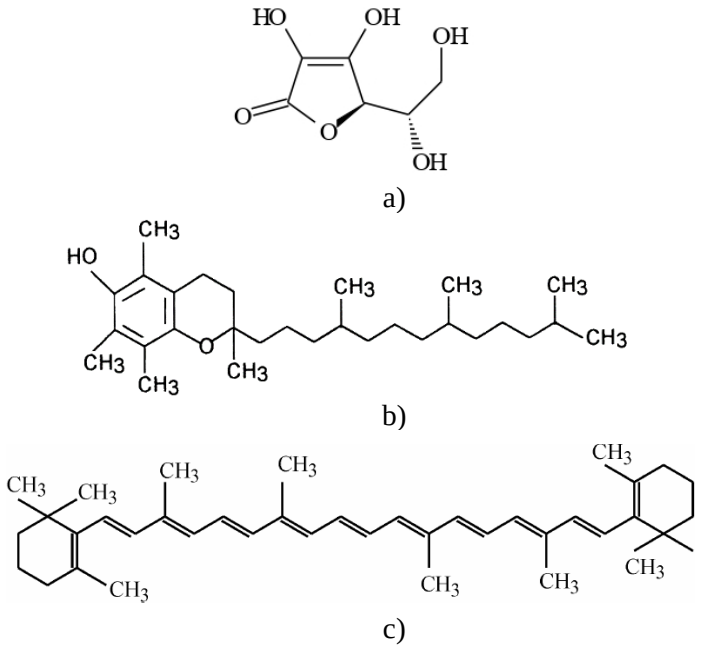

Fig. 2 – Chemical structures. a) ascorbic acid, b) tocopherol and c) β-carotene.

For example, in the literature it is emphasized that flavonoids from hot peppers have the highest antioxidant activity because there are many hydroxyl groups in their molecule, responsible for neutralizing free radicals (Capcanari *et al*., 2010).

Hot peppers could be harvest fresh all year and can be processed to extract the active compounds by various methods, namely: dehydration, freezing, sugar preservation, oil and vinegar maceration, as showed in a previous study (Cobzaru *et al*., 2019). Moreover, it has been noted that, after 4 years of preservation, the hot pepper oil extract is stable over time and to the action of UV radiation (Cobzaru *et al*., 2023), which makes it possible to use it in obtaining various cosmetic products or power supply even after a long time.

Considering the aspects presented above, this study attempted to identify capsaicin from the alcoholic and oily extract of hot peppers using FTIR spectroscopy. The antioxidant activity of the alcoholic hot pepper extract was also determined by the spectrophotometric method using 2,2-diphenyl-1-picrylhydrazyl (DPPH). Both the oily and alcoholic extracts were preserved for 5 years. By this we want to demonstrate the fact that, even after preservation for a long period of time the alcoholic extract of hot peppers retains its antioxidant activity (both through the existence of capsaicin and the test performed), and can be used to significantly increase the antioxidant properties of products cosmetics and food, where it will be added.

2. Experimental

2.1. Obtaining extracts for analysis

The hot pepper oil extract was obtained and presented in a previous study (Cobzaru *et al*., 2019). It was stored in a tightly closed glass container for 5 years in a dark and cool place.

The alcoholic extract of chili peppers was obtained by contacting chili peppers with 70-degree ethyl alcohol, for 14 days, in a ratio pepper: alcohol of 1:10 in a sealed glass container. Next, the mixture was filtered and the extract was stored in a tightly closed glass container in a dark and cool place. It should be noted that the alcoholic extract of hot pepper was also stored for 5 years, as was the oil extract. Both plant material, the oil and ethyl alcohol used for the study were purchased from the supermarket.

2.2. Analysis of hot pepper extracts

FTIR analysis was performed on an FTIR spectrometer (IR-Prestige, Shimadzu Europa GmbH, Duisburg, Germany), equipped with a DLATGS detector. The spectral resolution was 4 cm^{-1} and 128 scans were averaged for each spectrum. The ATR fixture used consists of a multi-reflection (10 reflections)

ZnSe plate (Pike Technology, Madison, USA). As a reference, the background spectrum of the air was recorded. The samples were measured directly on the ZnSe ATR crystal without the need for sample preparation beforehand. Between measurements the ATR crystal was cleaned with distilled water and then dried.

The antioxidant activity of the alcoholic extract of hot pepper was based on the ability of the compounds with antioxidant action to reduce the free radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) to diphenylpicrylhydrazine with the colour shift from purple to yellow and the decrease in absorbance recorded in the spectrophotometer proportional to the activity of the analysed sample (Wangensteen *et al*., 2004). For this purpose, a methanol solution of DPPH was prepared so that the absorbance recorded at 517 nm had a value between 1.1-0.9 absorbance units. 2950 µL of DPPH solution was added to the spectrophotometer cuvette and the initial absorbance (A_i) at 517 nm was read. 50 µL sample was added, homogenized and the absorbance (A_f) reread after 3 minutes. The percentage of free radical inactivation (equivalent of antioxidant activity) was calculated taking into account the decreased value recorded for absorbance. In parallel, the antioxidant activity of the solvent used for extraction was also analysed in order to evaluate the influence of the solvent on the antioxidant capacity of the sample.

3. Results and Discussions

1. Extraction of capsaicin from canned hot pepper samples

Figure 3 shows the FTIR spectrum of the hot pepper oil extract preserved for 5 years.

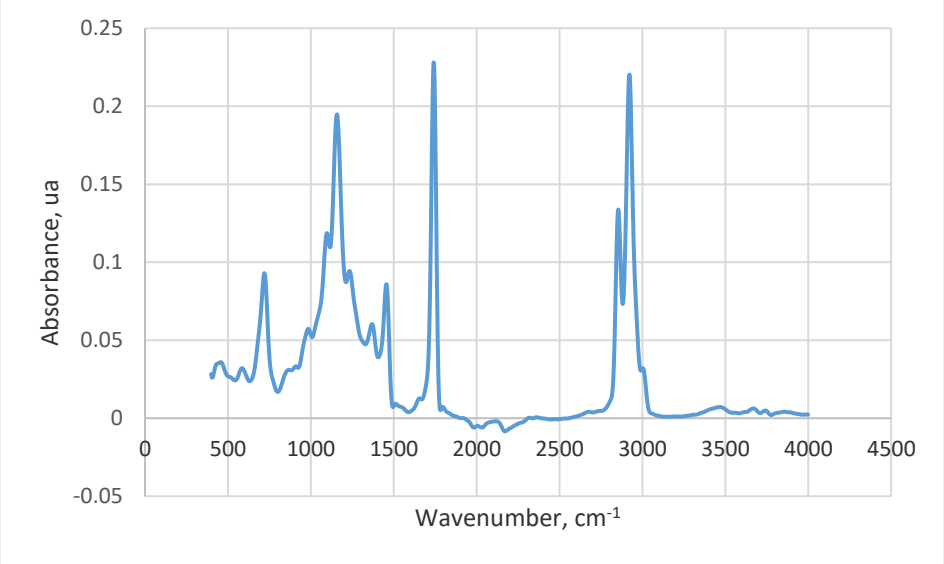

Fig. 3 – FTIR spectrum of hot pepper oil extract preserved for 5 years.

In Fig. 3, it can be seen that the FTIR spectrum contains characteristic absorption bands for fatty acids, the constituents of vegetable oils. In addition to these, it can be observed that the FTIR band is dominated by a very intense stretching vibration characteristic of the C=O bond at 1744 cm^{-1}, while the deformation vibrations specific to the C-H bond are present at 1466 cm^{-1}, and a of C-C and C-O bonds are present at 1097 and 1161 cm^{-1}, respectively.

Figure 4 shows the FTIR spectrum of the alcoholic extract of hot peppers preserved for 5 years.

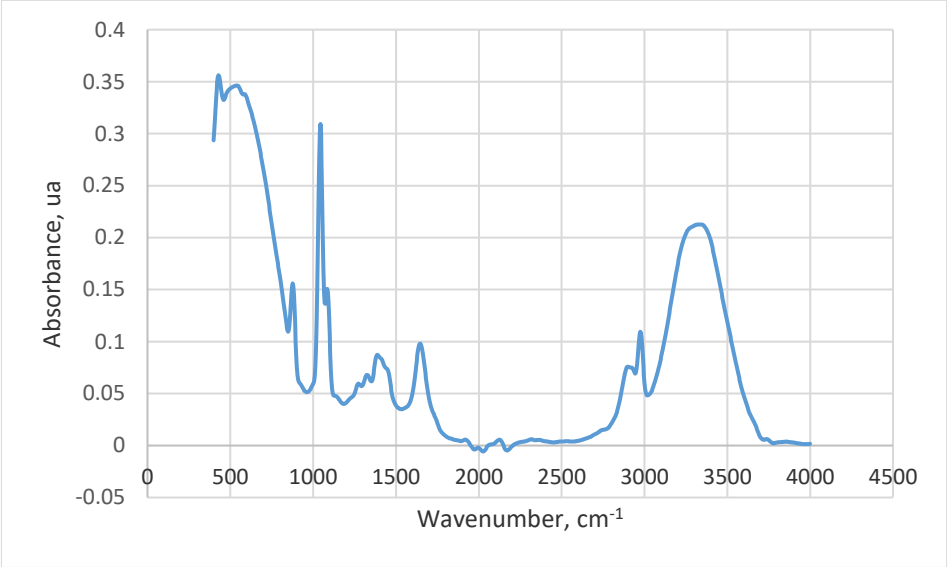

Fig. 4 – FTIR spectrum of the alcoholic extract of hot peppers preserved for 5 years.

From Fig. 4 it can be seen that the FTIR spectrum contains a band characteristic of the broadened N-H and O-H bond that can be observed at 3447 cm^{-1}, and 1633 cm^{-1}, 1283 cm^{-1} and 806 cm^{-1} belong to the characteristic bands of C bonds =O, C-N and C-H.

Therefore, the F-TIR spectra in Figs. 3 and 4 show characteristic bands for the specific bonds in the structure of capsaicin from the hot pepper, which are also confirmed by the literature (Peng *et al*., 2015). In addition to these, a series of peaks that have not been identified and that could belong to the other existing compounds in the composition of hot pepper can be observed. However, due to the weak polarity of the bonds, the FTIR absorption bands are relatively weak, and therefore for a more accurate qualitative and quantitative evaluation, the implementation of chemometric algorithms is needed

The study on the antioxidant activity of the alcoholic extract of hot pepper showed that its value is 46.73%, falling within the range of 20-66%, specified in the specialized literature (Capcanari *et al*., 2010). This proves that the alcoholic extract, even after a long preservation time (5 years), can be used in the

preparation of various cosmetic or food products to improve their antioxidant properties and have a positive impact on the body

4. Conclusions

In this study, a study to identify capsaicin from the alcoholic and oily extract of hot pepper using FTIR spectroscopy was made. The spectra obtained show characteristics band that confirm the presence of functional groups specific to capsaicin from hot pepper. However, due to the weak polarity of the bonds, the FTIR absorption bands are relatively weak, and therefore for a more accurate qualitative and quantitative evaluation, the implementation of some chemometric algorithms is needed.

The antioxidant activity of the alcoholic extract of hot pepper was also determined. The study on the antioxidant activity of the alcoholic extract of hot pepper by the spectrophotometric method using 2,2-diphenyl-1-picrylhydrazyl (DPPH) showed that its value is high, which proves that the alcoholic extract, even after a long preservation time (5 years) can be used in the preparation of various cosmetic or food products to improve their antioxidant properties and have a positive impact on the body.

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IDENTIFICAREA CAPSAICINEI DIN EXTRACTUL ALCOOLIC ȘI ULEIOS DE ARDEI IUTE CU AJUTORUL SPECTROSCOPIEI FTIR

(Rezumat)

În acest studiu s-a încercat identificarea capsaicinei din extractul alcoolic și uleios de ardei iute cu ajutorul spectroscopiei FTIR. Atât maceratul uleios cât și alcoolic au fost conservate timp de 5 ani. Spectrele obținute prezintă diferite caracteristici ale benzii care confirmă prezența unor grupări funcționale specifice capsaicinei din ardeiul iute. De asemenea, s-a determinat activitatea antioxidantă a extractului alcoolic de ardei iute prin metoda spectrofotometrică care utilizează 2,2-difenil-1-picrilhidrazil (DPPH). Chiar și după o perioadă îndelungată de conservare, extractul alcoolic de ardei iute are o activitatea antioxidantă mare, putând fi adăugat în prepararea produselor cosmetice și alimentare pentru a le crește semnificativ proprietățile antioxidante și a avea un impact pozitiv asupra organismului.