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Microwave assisted extraction of benzofurane derivative from *Petasites hybridus rhizomes*

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Abstract: Extraction of benzofuran derivative of *Petasites hybridus* rhizomes from north of Iran was performed by Microwave instrument and by mixing of two polar solvents such as methanol or tetrahydrofuran and water. After extraction and purification, the structure of this compound was assigned by ^{1}H and ^{13}C NMR spectroscopy and confirmed by X-Ray diffraction analysis.

Keywords: ^{1}H and ^{13}C NMR Technique, Petasites plant, Rhizomes, Polar solvents, Microwave-assisted.

Introduction

Highly substituted furans play an important role in organic chemistry, not only as key structural units in many natural products, common subunits in pharmaceuticals [1-7], fragrances [8], and flavors [9], but as useful building blocks in synthetic chemistry [10-14]. They have also found utility as synthetic intermediates or synthons for numerous functional groups; *inter alia*, carboxylic acids, α-keto-esters, and aromatics [15]. For this reason, the efficient synthesis of multiply substituted furans continues to attract the interest of synthetic chemists [16, 17]. One of the sources of this important compound is a type of plant from the north of Iran. It is mentionable that these classes of compounds have marvelous biological and medicinal properties and are used in treatment of a bad type of migraine and MS disease [20]. Phytochemical analyses of extracts of *P. hybridus* reveal are very different patterns as a medicine compounds and a few papers were observed about this type of plant [18-21]. Hence, in this research, we have used Microwave-irradiation as green source energy for extraction of benzofuran derivative from rhizomes of *Petasites hybridus* [22-26].

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Results and discussion

As part of our current studies on the development of new routes in heterocyclic synthesis, we report an efficient route for preparation of benzofuran derivative. Herein, for extraction of benzofuran derivative (Figure **1**), the powder form of Petasites hybridus plant was placed in a run-bottom flask with reflux system and Microwave-irradiation.

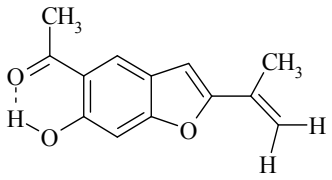

Fig. 1: Structure of extracted benzofuran derivative

In this method many kind of polar solvents were tested, but the best solvent was mixture of methanol or tetrahydrofuran and water (50:50). The yield of this

process in mixture of methanol or tetrahydrofuran and water is a more than when water is used as the solvent. If extraction is performed in organic solvents without Mw energy source, it takes long time (at least 20 h) and some time the extracted compound is decomposed if the mixture is heated over 50°C. Because of this reasons, we used MW instrument (*MICROSYNTH* from Mylestone Company) [27]. The short time and good yield of extraction are advantages of this process [28, 29]. Therefore, extraction was performed in mixture of methanol or tetrahydrofuran and water (50:50) as solvent by using of MW in normal pressure and at below 50 °C at about 40 mines on the power of 500 W.

By employing of Mw energy about 40-60 min, a yellow extracted compound was observed in solvent media. By removing of solvents under reduced pressure, a yellow sticky compound was reminded in the bottom of the flask that was recrystallized in mixture of THF and ethanol (2:1). After extraction and purification this compound, the structure of that was assigned by ^{1}H and ^{13}C-NMR spectroscopy and confirmed by X-ray diffraction analysis (Figure **2**).

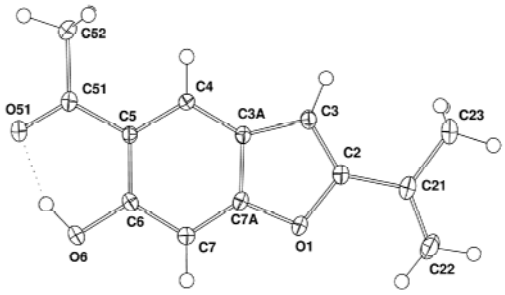

Fig. 2: X-ray structure of yellow extracted benzofuran derivative

The ^{1}H NMR spectrum of extracted compound exhibited nine sharp singlet signals which are arised from two methyl (δ = 2.09 and 2.67 ppm), two geminal methin (5.17 and 5.74 ppm), two methin for aromatic moiety (6.53 and 7.89), one methin for furan moiety (6.96 ppm) and OH (12.49 ppm) protons. Hydrogen of OH because of hydrogen bonding with oxygen of carbonyl group was appeared in low magnetic field (12.49 ppm).

In conclusion, we have reported an efficient approach for extracting of benzofuran derivative from rhizomes of *Petasites Hybridus* in North of Iran. This extraction was performed by microwave energy in mixture of methanol or tetrahydrofuran and water (50:50). After extraction process, the crude extract was purified and recrystallaized in special polarity of solvent. For future time, consideration of biological activation of the extracted compound is a main topic for research work.

Experimental

Material and methods

All solvents were obtained from commercial sources. Melting points were measured on a Kofler hot stage apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were obtained with a Bruker FT-500 spectrometer in chloroform-d1, and tetramethylsilane (TMS) was used as an internal standard. X-ray diffracted intensities were measured from single crystals at 100 K on an Oxford Diffraction Gemini-R Ultra CCD diffractometer using monochromatized Cu-K_{α}) (λ= 1.54178 A°).

Representative procedure for extracted of benzofuran derivative.

After cleaning and cutting of rhizomes of Petasites hybridus (very small), we have added methanol or tetrahydrofuran and water (50:50) and the mixture was heated for 40-60 min. After this time, a yellow extracted compound was observed in solvent. By removing of solvents under reduced pressure, a yellow needle crystal (benzofuran derivative) was formed in 75% yield.

1-(6-hydroxy-2-isopropenyl-1-benzofuran-5-yl)-1-ethanon

Yellow crystals, mp 116-118°C. ^{1}H NMR: δ = 2.09 (3 H, s, Me), 2.67 (3 H, s, Me), 5.17 (1 H, s, CH), 5.74 (1 H, s, CH), 6.53 (1 H, s, CH), 6.96 (1 H, s, CH), 7.89 (1 H, s, CH), 12.49 (1 H, s, OH) ppm. ^{13}C NMR: δ = 19.2 (Me), 26.7 (Me), 99.4 (CH), 102.4 (CH), 113.7 (CH_2), 116.8 (C), 121.9 (C), 123.4 (CH), 132.1 (C), 157.9 (C), 159.7 (C), 161.6 (C), 203.8 (C=O) ppm.

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