



The effect of microwave pretreatment on some physico-chemical properties and bioactivity of Black cumin seeds' oil

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ABSTRACT

In the current study, different processing times including 90, 180 and 270 s and microwave powers including 180, 540 and 900 W were applied for optimizing of the extraction process. After microwave pre-treatments, the oil seeds were extracted with screw press with different rates (11, 34 and 57 rpm), then parameters including extraction efficiency, oxidative stability, peroxide and acidity index, DPPH free radical scavenging activity as well as the refractive index of the extracted oil were studied. Statistical analysis and process optimization was performed with the use of *response surface methodology (RSM)*. The results revealed that enhancement in the microwave power and the processing time increased extraction efficiency, acidity index and oil peroxide value, but it decreased the oxidative stability value of the achieved oil. The achieved results also showed up that the studied parameters had no significant impacts on the refractive index; moreover the extraction efficiency was reduced with an enhancement in the rotational rate of the screw press. According to the process optimization results, it might be stated that with applying processing time for about 185.44 S, microwave pretreatment of 718.65 W and screw-rotation speed of the press of 11 rpm, the desired outcomes are reached.

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1. Introduction

One of the basic essential foods in world population growth in is extracting oils from economic and healthful plants resources, not only it requires in nutraceutical industries but also it is required to be applied in pharmaceutical and cosmetic industries revealing the importance of oil seeds. Expanding of researches on this field in Iran seems very essential since almost 94% of the oil seeds is imported from overseas (Iran Nezhad and Hoseini Mazinani, 2017). *Nigella sativa* L. (Ranunculaceae family) is an annual herbaceous plant whose growth area extends from the countries of the southern and eastern-rim of the Mediterranean basin to Iran, Pakistan and India. In the ethno-pharmacology fields of the abovementioned regions (countries), *Nigella sativa* L. seeds due to containing considerable quantities of compounds with bioactivities are used in treatment of a wide range of diseases including gastro-intestinal as well as skin and respiratory ailments (Farzaneh and Carvalho, 2015; Riaz et al., 1996). Moreover Black cumin seeds due to hav-

ing hot or spicy characteristics have been using for centuries in culinary arts of different cultures (Ramadan, 2007). Along with the all abovementioned advantages the plant's seeds is considered as one of the novel sources of edible oils due to having considerable quantity of oils as well as oils with many health properties (Cheikh-Rouhou et al., 2007). Both seeds and oils are often used as nutritional supplement due to its various health properties, particularly anti-inflammation, antioxidant and as a result anti-tumor activities (Ghosheh et al., 1999; Houghton et al., 1995), antibacterial activities (Morsi, 2000) as well as having a stimulatory impact on the immune system (Salem and Hossain, 2000) for this plants are reported. Finally, *N. sativa* L. seeds were used in preparation of a highly scored nutritive oil. Although in the world scale *Nigella* seeds oil have really no considerable impacts on economic market, it nevertheless constitutes a niche market whose size is constantly growing due to its considerable pharmacological properties as well as religious causes because it is frequently used in sacred books, moreover *Nigella* seeds in Tutankhamen tomb have been found (Padhye et al., 2008). More studies have reported this seed's proximate contents for moisture, oil, protein, ash and total carbohydrate in the following range 3.8–8.65%, 24.48–40.35%, 20.8–26.7%, 3.7–4.86% and 24.9–40.0%, respectively regarding the

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abovementioned order (Bassim Atta, 2003; Cheikh-Rouhou et al., 2007; Takruri and Dameh, 1998). Minerals found predominantly in this seed are reported as the following: potassium, calcium, phosphorus and magnesium (Sultan et al., 2009). Extracted oil is abundant in oleic, linoleic and linolenic acids which are unsaturated fatty acids with many recorded potential health benefits (Bassim Atta, 2003; Farzaneh and Carvalho, 2015).

Solvent extraction assisted by sonication and stirring might also be time consuming, and requires huge volume of solvents that are sometimes hazardous and requires more steps in concentration (Rajaei et al., 2008). However, the uses of solvent have some disadvantages such as long-time process, high costs, safety issues, eventual emissions of volatile organic compounds into the environment and low quality of the products extracted through processing at high temperatures (Anderson, 1996). Oil extraction efficiency with hot pressing is higher than cold pressing, but due to the heat generated during the compressing, the quality of the resulting oil is lower, the oil extracted by cold pressing preserves its natural properties and is free of chemical materials; therefore, the demand for oils obtained by cold pressing approach is getting increased (Siger et al., 2008). In the oil extraction with the use of cold pressing method, different parameters including the pressing pressure, seed moisture and processing temperature might affect on the efficiency of the extracted oils. In this respect, recently novel extraction techniques with the use of supercritical solvents (Meireles, 2003), ultrasound (Lou et al., 2010), microwave treatments (Taghvaei et al., 2014) and alternating electric fields (Zeng et al., 2010) have been taken into consideration. Among these emergent technologies, microwave assisted extraction (MAE) has revealed many advantages such as convenience, less processing times, and high efficiency (Flamini et al., 2007; Zhai et al., 2009). MAE uses microwave radiation as a source of heat for the solvent-sample mixture. Due to the particular impacts of microwaves on matter (namely dipole rotation and ionic conductance), heating with microwaves is instantaneous and occurs in the interior of the sample, leading to rapid extraction (Camel, 2001). The main advantage of MAE is its internal heating mechanism (Périno-Issartier et al., 2011); acceleration of the extraction could be partly explained by the specific impacts of microwaves on plant materials (Camel, 2001). Response surface methodology (RSM) along with artificial neural network (ANN) is a useful and well known statistical technique in modelling and optimizing of different processes (Farzaneh et al., 2016; Ghodsvai et al., 2016; Rostami et al., 2014; Vieira et al., 2012). This method is a set of statistical techniques which could evaluate how the intended responses are affected by several independent variables (Manivannan and Rajasimman, 2008). Optimum oil extraction conditions with microwave as a pretreatment varies with the nature of the meal, the solvent type used etc. For instance, maximum oil yields from *Scenedesmus obliquus* was recovered with the use of microwaves at 95 °C after 30 min reaction time (Balasubramanian et al., 2011). Optimum oil yields of rice bran and soybean oils were obtained at 73 °C and reaction times of 21 and 17 min, respectively with the application of microwave (Terigar et al., 2010). Optimised microwave extraction conditions for olive oil were determined to be 17 min for extraction time, 17 min for leaching time in 720 W microwave power (Viot et al., 2007). A group of scientists studied the impacts of temperature and the proportion of solid/solvent on the efficiency of the ultrasound assisted extraction of Black cumin with the use of response surface methodology (RSM). The optimised extraction conditions were achieved as the following: extraction temperature of 32 °C, the proportion of solid/solvent equals to 1/20 within the extraction time of 30 min. An enhancement in the proportion of solid/solvent and ultrasound pretreatment time resulted, in an enhancement in the quantity of the extraction in oil emission stage, while an enhancement in temperature without ultrasound assist, resulted

in the enhancement in oil extraction in washing stage. The study on the kinetics of oil extraction revealed that ultrasound waves might improve the coefficient of mass transfer in washing and emission stages in comparison to the extraction process without ultrasound assist (Abdullah and Koc, 2012). Since no researches on the optimization of oil extraction from Black cumin seeds with the use of microwave pretreatment were found by the authors, in the present study, it was attempted to examine and optimise the influence of effective parameters in ultrasound assisted extraction with the use of response surface methodology (RSM) tool. Moreover the impacts of different parameters including the microwave time and power as well as screw rotational speed on the physico-chemical, antioxidant and oxidative stability properties of Black cumin extracted oil was studied. The aim of this research is discovering of novel application in extraction of oils from oil seeds with high efficiency and quality. Since within oil extraction huge quantity of the oil remains in cells therefore application of the optimised conditions for the extraction seems very effectual for both industries and human nutrition.

2. Materials and methods

2.1. Materials

2.1.1. Plant materials

Fresh Black cumin seeds with the scientific name of *Nigella sativa* L. (Ranunculaceae family) used in this research were bought from local market of Gonbad-e-Qavus city-Golestan province. The primitive moisture of the samples was detected (9.5%). The fresh seeds were dried in shadow in room temperature, after they dried completely; the seeds were shifted to plastic bags and sealed tightly with the use of vacuum sealer. Afterward the sealed samples were shifted to refrigerator and preserved until the experiments day.

2.1.2. Chemical and reagents

The chemical materials including hydroxide sodium, phenolphthalein, ethylic alcohol, methyl alcohol, sodium thiosulfate, chloroform and acetic acid were purchased from Merck Company. DPPH, starch and Potassium iodide were provided from Sigma Aldrich. All reagents were provided of analytical grade.

2.1.3. Equipments

Equipments used in this research are as the following: spectrophotometer (Biochrom, UK), laboratory sieve, Rancimat device (Metrohm, Switzerland), desiccator, laboratory oven (Memert, Germany), centrifuge (Thermo, Japan), Scanning Electron Microscopy (SEM) (S-360, Oxford., England), digital scale (Gec Avery, UK), microwave (LG, South Korea) and laboratory screw press (Kern Kraft, Germany) have been used in this research.

2.2. Methods

2.2.1. Sample preparation and oil extraction

In the current study, after preparation of the samples, black cumin seeds were cleaned and sieved to remove the broken seeds and stones as well as further external materials, afterward to were transferred and preserved in plastic bags resistant to the moisture and air flow until the experiments. Moreover a part of the sample was removed and the primitive oil content of the sample was determined with the use of soxhlet system (40.4%). Then, the cleaned samples were shifted and exposed under microwave pretreatments within different processing times (90, 180 and 270 s) and microwaves powers (180, 540 and 900 W) (Kittiphoom and Sutasinee, 2015). Afterward, the seeds' oil was extracted with screw

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