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Impact of amine- and phenyl-functionalized magnetic nanoparticles impacts on microwave-assisted extraction of essential oils from root of *Berberis integerrima* Bunge

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ABSTRACT

In this study, essential oils were extracted from the roots of *Berberis integerrima* Bunge using hydrodistillation (HD) and modified microwave-assisted hydrodistillation (MAHD) techniques for the first time. In the first step, amine- and phenyl-functionalized magnetic nanoparticles were synthesized through the silanization method. Afterwards, their effects on extraction efficiency were investigated from quantitative and qualitative points of view. The results obtained from the conventional HD method explicitly showed that the quantity of the extracted essence was negligible both in the presence and absence of modified synthesized nanoparticles but, the extraction yield was significantly increased through the MAHD method, particularly in the presence of the modified magnetic nanoparticles, as extraction yields were 0.16, 0.61 and 0.71 w/w% and chemical diversity of 10, 10 and 18 compounds were obtained from MAHD, MAHD with modified anyl, and with modified phenyl magnetic nanoparticles, respectively.

1. Introduction

The evergreen and self-fertile genus *Berberis* belonging to family Berberidaceae is among the well-known genera because of its diversity and pharmacological uses in traditional and folk medicine since ancient times. *Berberis* is mainly distributed in the Northern Hemisphere, primarily in the Himalayan region (Bhardwaj and Kaushik, 2012). Barberry fruits are widely used as food flavouring agents (Font et al., 1993). It is used in the treatment of gastrointestinal disease, bleeding, swollen gums and infected teeth, sore throat, fever, bile, malaria, hepatitis, inflammation, and diarrhea. In addition, it plays an important role in reducing blood serum cholesterol. Berberine is an effective agent to reduce triglyceride and low-density lipoprotein (LDL) with dyslipidemia in animals (Ashraf et al., 2013).

In view of the significance of the genus *Berberis*, numerous researchers have explored the phytochemical and pharmacological aspects related to its different species worldwide. In the literature, sporadic reports are found concerning the anticonvulsant (Bhutada et al., 2010); antihyperglycemic and antihyperlipidemic (Ashraf et al., 2015); antibabesial (Elkhateeb et al., 2007); antihypertensive and vasodilator (Fatehi-Hassanabad et al., 2005); anticoccidial (Malik et al., 2014); antioxidant, anti-inflammatory, and anti-acetylcholinesterase (Miguel et al., 2014); antihistaminic and anticholinergic (Shamsa et al., 1999); antibacterial (Villinski et al., 2003); antimicrobial hepatoprotective (El-Salam et al., 2015); and anticoagulant (Elyas and Razieh, 2011) activities and properties of this genus. In view of the many activities of the plants belonging to the *Berberis* genus, the isolation, characterization, and determination of their essential oils and volatile components seem highly justified.

Various methods can be used for the extraction of plant active materials which include steam distillation, Soxhlet extraction, and solvent extraction (Kovacevic and Kac, 2001; Yu et al., 2004). However, appreciable loss of certain volatile compounds, low extraction efficiency, and toxic solvent residue in the extract may be encountered when these separation methods are used. Furthermore, these extraction procedures are time-consuming and uneconomical. These disadvantages result in the consideration of new techniques like microwave-assisted extraction (MAE), that uses less solvent, time, and energy for the extraction of essential oils.

MAE is suitable for the recovery of a large array of compounds; it has been recognized as a versatile and efficient extraction technique for isolation of essential oils from a large number of plants. Compared with classical reflux and Soxhlet-based extractions, MAE generally shows evident advantages that involve shorter extraction time, higher

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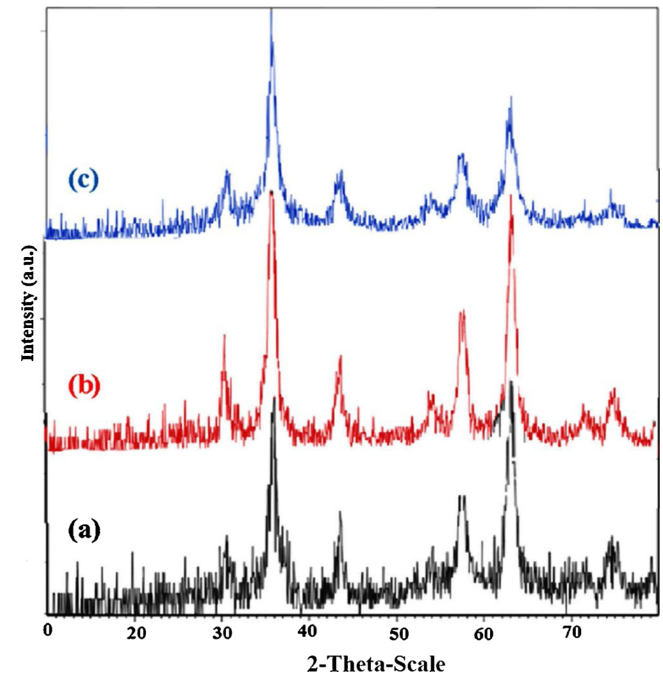

Fig. 1. XRD image of (a) non-modified magnetic nanoparticles, (b) AFMN, and (c) PFMN.

extraction yield, higher selectivity, and better quality of target extracts (Hashemi-Moghaddam et al., 2015). In addition, this technique is more cost-effective than accelerated solvent extraction (Lucchesi et al., 2004; Zhang et al., 2011; Mohammadhosseini et al., 2013). Traditionally, MAE utilizes a distinct volume of an organic solvent such as methanol. Moreover, extraction efficiency of microwave-based techniques may be considerably poor when either the target compounds or solvents are non-polar, or when the viscosity of solvent is extremely high (Sahena et al., 2009). In general, the MAE strategy is not recommended for the extraction of thermally labile compounds (Zhang et al., 2011)

In the present study, a simple, modified magnetite nanoparticle-assisted microwave distillation method is proposed for the effective extraction and analysis of the essential oils from *Berberis integerrima* Bunge roots in single factor experiments. Accordingly, amine- and phenyl-functionalized magnetite nanoparticles practically served as the microwave absorption solid medium. This approach can be considered as a feasible way to isolate essential oils from a wide variety of plant organs. Evidently, the types of radiation-absorbing materials should have promising microwave absorption capacities in this separation methodology (Kim et al., 2005).

To the best of our knowledge, this report is the first concerning the characterization of essential oils from roots of *B. integerrima* Bunge, an herbal and medicinal plant that grows wild in Iran. The lack of adequate reports highlighting the chemical composition of the essential oils using synthesized amine- and phenyl-functionalized magnetic nanoparticles prompted us to conduct this study. This study confirmed the high capability and effectiveness of the modified magnetic nanoparticle-based method for the separation of essential oils in a microwave oven.

2. Materials and methods

2.1. Materials

2.1.1. Plant material

B. integerrima Bunge was harvested on September 20, 2015 from Damghan, Badelekoh region, Semnan Province, northeast Iran (Latitude:36° 23′ 37.916″ and Longitude:53° 57′ 12.833″) at an altitude of 2462 m above the sea level. The plant was deracinated and its root was separated; polyethylene gloves were worn to prevent unwanted contamination. After sampling, the plant was shipped on wet ice and arrived in the laboratory 14 h later. Thereafter, the plant was identified by a local botanist, and a voucher specimen (number 445200) was deposited at the herbarium of the Botanical Department of Islamic Azad University, Damghan Branch, Iran. The plant roots were dried at shadow with particular care to avoid additional damage and minimize cross-contamination. Moisture content of fresh samples was calculated after oven drying for 48 h at 60 °C. The average moisture content was 46.7%.

2.1.2. Chemicals

Fe_2O_3 nanoparticles were purchased from Sigma Aldrich, 3-aminopropyltriethoxysilane (APTTS), phenyltriethoxysilane, ethanol, and *n*-hexane were all purchased from Merck (Darmstadt, Germany).

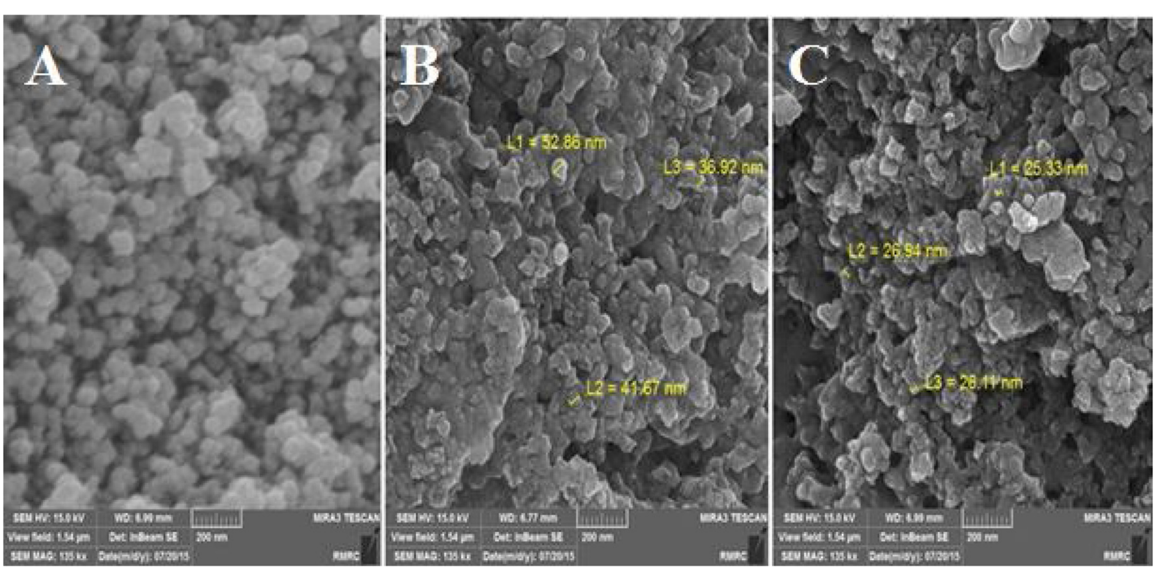

Fig. 2. Scanning electron microscopy images of non-modified and modified magnetic. nanoparticles under 135,000 magnifications: (a) magnetic nanoparticles, (b) AFMN, and (c) PFMN.