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Research article

Canola oil is an excellent vehicle for eliminating pesticide residues in aqueous ginseng extract

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ABSTRACT

Background: We previously reported that two-phase partition chromatography between ginseng water extract and soybean oil efficiently eliminated pesticide residues. However, an undesirable odor and an unpalatable taste unique to soybean oil were two major disadvantages of the method. This study was carried out to find an alternative vegetable oil that is cost effective, labor effective, and efficient without leaving an undesirable taste and smell.
Methods: We employed six vegetable oils that were available at a grocery store. A 1-mL sample of the corresponding oil containing a total of 32 pesticides, representing four categories, was mixed with 10% aqueous ginseng extract (20 mL) and equivalent vegetable oil (7 mL) in Falcon tubes. The final concentration of the pesticides in the mixture (28 mL) was adjusted to approximately 2 ppm. In addition, pesticides for spiking were clustered depending on the analytical equipment (GC/HPLC), detection mode (electron capture detector/nitrogen–phosphorus detector), or retention time used. Samples were harvested and subjected to quantitative analysis of the pesticides.
Results: Soybean oil demonstrated the highest efficiency in partitioning pesticide residues in the ginseng extract to the oil phase. However, canola oil gave the best result in an organoleptic test due to the lack of undesirable odor and unpalatable taste. Furthermore, the qualitative and quantitative changes of ginsenosides evaluated by TLC and HPLC, respectively, revealed no notable change before or after canola oil treatment.
Conclusion: We suggest that canola oil is an excellent vehicle with respect to its organoleptic property, cost-effectiveness and efficiency of eliminating pesticide residues in ginseng extract.

1. Introduction

The pesticide strategy concerns *plant protection products*, which are those pesticides that are used to protect plants and plant products from pests, diseases, and weeds and to regulate the growth of plants [1]. It is generally known that crop yields will drop by more than 30% without the use of pesticides. In this case farmers have to expand their fields to compensate for the decrease in crop yield. To increase the field size, forests have to be destroyed, which can be detrimental to the environment. Pesticides help farmers protect their crops from pests, fungi, and weeds so that people can enjoy an abundance of high quality food. However, this food must be safe to eat. Careful use of pesticides can deliver substantial benefits for society: increased availability of good quality crops; reasonably priced foodstuffs, in particular, fruits and vegetables; and clean urban environments. However, pesticides can, by their nature, be harmful to living organisms, so there are risks associated with their use. It is important that these risks are accurately assessed and that appropriate measures are taken to minimize them [2]. However, some pesticides, specifically lipophilic agents:

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are not biodegradable; accumulate in soil, at the water surface, in plants, and in the bodies of animals and shellfish; and are recycled via the food chain, which in turn harms humans. Due to their toxicity, the use of some pesticides is strictly restricted or forbidden.

Crop cultivation without pesticides seems almost impossible, particularly for ginseng because it should be cultivated for 6 years. It is well known that the loss of its root due to phytopathogens and insects accounts for a 10% crop loss every year, indicating that there is > 50% loss of ginseng crop over 6 years [3]. Chemical pesticides are environmentally unfriendly due to their physicochemical properties and recalcitrance to biodegradation. For example, chemicals, such as the dichlorodiphenyltrichloroethane (DDT) metabolite dichlorodiphenyldichloroethylene, are termed endocrine disruptors, which are known to elicit adverse effects by mimicking or antagonizing natural hormones in the body. A long-term, low-dose exposure of these pesticides has been linked to human health effects, such as immune suppression, hormone disruption, diminished intelligence, reproductive abnormalities, and cancer [4–6]. Therefore, DDT derivatives were forbidden to be manufactured and used almost half a century ago. However, DDT derivatives are still detected in the soil of ginseng fields. In addition, the soil environment is interconnected from country to country because of atmospheric circulation. Thus, it seems necessary to develop a health-friendly method of eliminating pesticide residues or environmentally unfriendly chemicals after harvest.

There have been many attempts to develop an elimination method for pesticide residues, including microwave decomposition, photolysis, and CO_2 supercritical extraction methods. However, most of the methods have poor effectiveness in view of the efficiency of elimination, loss of active ingredients of ginseng, labor, and, most importantly, cost. Among them, the CO_2 supercritical extraction method has a greater advantage in the efficiency of pesticide residue elimination compared to the other methods. However, importantly, it has a disadvantage in its capacity for treatment per unit time and cost-effectiveness [7,8].

We have developed a partition chromatography method for removing residues using soybean oil as a solvent. It has great advantages over the other methods with respect to cost, labor, and efficiency [9]. However, it gives an unpalatable smell and taste to the soybean oil-treated ginseng extract. Therefore, a new vegetable oil that is devoid of undesirable taste and smell but is effective in terms of cost, labor, and efficiency has to be developed. We investigated the pesticide residue elimination efficiency, change in ginsenosides profiles, and smell and taste before and after treatment of the ginseng extract with six vegetable oils that are readily available in the market.

We propose a new cost effective, labor effective, and efficient vegetative oil extraction method of removing pesticide residues from ginseng extract that is based on two-phase partition chromatography between six different oils and aqueous ginseng extract as well as organoleptic tests.

2. Materials and methods

2.1. Chemicals and materials

Pesticide standards were purchased from Chem Service (West Chester, PA, USA), and six different vegetable oils were purchased from a local grocery store. Ethanol (70%) ginseng extract devoid of pesticide residues was prepared by our laboratory. A LC-Florisil solid-phase extraction tube for pesticide purification was purchased from Supelco (St Louis, MO, USA). Silica gel TLC employed for the qualitative analysis of ginseng saponin was procured from Merck (Darmstadt, Germany). The organic solvents employed for the quantitative analysis of ginsenosides were HPLC grade (Tedia, Fairfield, OH, USA). Fourteen reference ginsenosides [Rb1, Rb2, Rc, Rd, Re, Rf, Rg1, Rg2(*S*), Rg2(*R*), Rh1, Rh2(*S*), Rh2(*R*), Rg3(*S*), Rg3(*R*)] were kindly supplied by the Korea Ginseng Corporation (Seoul, Korea).

2.2. Equipment for chemical analysis

An Agilent 6890N gas chromatography device was used for the analysis of pesticide residues. The detector temperature was set to 300°C, the H_2 flow rate was 3.0 mL/min, the air flow rate was 60 mL/min, and there were columns in the HP-5MS capillary column (30 m × 0.25 mm, 0.25 μm, Agilent) and DB-17MS capillary column (30 m × 0.25 mm, 0.25 μm, Agilent). The gas flow rate was 1.0 mL/min. The HPLC device used for the analysis of pesticide residue was a Hewlett Packard 1100 (Agilent, Santa Clara, CA, USA). The detection wavelength was set to 254 nm and 275 nm and the column was an InsertSustain C18 column (4.6 × 250 mm, 5 μm; GL Science, Torrance, CA, USA). The mobile phase was a mixture of water and acetonitrile: 70:30 (0–5.0 min) and 15:85 (5.0–22.0 min) with a flow rate of 1.0 mL/min.

2.3. Two phase-partition chromatography between vegetable oils and aqueous ginseng extract

The collection of pesticides used for spiking into the mixture of aqueous ginseng extract and vegetable oil were grouped depending on their retention time in the GC or HPLC to avoid overlapping their fingerprint: GC/electron capture detector (ECD) group 1, pentachloronitrobenzene, pentachlorothioanizole (PCTA), pentachloroaniline (PCA), tefluthrin, chlorothalonil, dichlorodiphenyldichloroethylene, endrin, dichlorodiphenyldichloroethane, cyfluthrin, and DDT; GC/ECD group 2, α-hexachlorocyclohexane (HHC), β-HHC, γ-HHC, δ-HHC, aldrin, dieldrin, bifenthrin, prochloraz, and difenoconazole; GC/nitrogen–phosphorus detector (NPD) group 1, tolclofos-methyl, diethofencarb, hexaconazole, flusilazole, and carbosulfan; and GC/NPD group 2, cyprodinil, flutolanil, buprofezin, kresoxim-methyl, tebuconazole, amitraz, and methalaxyl. HPLC analysis was carried out in one group under one condition but two different wavelengths (254 nm, 275 nm) were used for detection of acetamiprid, carbofuran, dimethomorph, fluquinconazole, pyrimethanil, cyazofamid, pyraclostrobin, and sethoxydim. A total of 1 mL of six different oils spiked with a predetermined amount of 32 pesticides (final concentration ca. 2 ppm for each) was mixed with the 10% ginseng extract (20 mL) and the corresponding oil (7 mL) in 50 mL Falcon tubes. The tubes were then vortexed and centrifuged at 3,000 rpm (1,500 x g) for 15 min. The lower aqueous layer was harvested with a Pasteur pipette, and both layers were subjected to pesticide analysis by GC or HPLC after purification by LC–Florisil column chromatography. The lower ginseng extract layer was further subjected to ginsenoside profile analysis by TLC and HPLC.

2.4. Analysis of multiresidue pesticides

Analysis of pesticide residues was carried out by the multiresidue methods described in the pesticide analytical manual [7]. All of the pesticides in the aqueous phase (25 mL) were extracted with CH_3CN (100 mL). The CH_3CN layer was harvested 1 h after NaCl (10–15 g) addition. The acetonitrile fraction was then dried *in vacuo* and passed through an LC-Florisil SPE tube after being dissolved in hexane containing 20% acetone. The eluate was concentrated *in vacuo* at a temperature below 40°C, dissolved in hexane (2 mL) containing 20% acetone and subjected to GC/ECD or GC/NPD. Pesticides in the oil phase (2 mL) were dissolved in hexane (5 mL) and partitioned with hexane-saturated CH_3CN (100 mL, 3 times). The CH_3CN fraction was then washed with 30 mL of CH_3CN-