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OPTIMISATION OF OIL EXTRACTION FROM HALOPHYTE SP. SEEDS

Carmen CONSTANTIN[1], Loubna HADHOUM[2], Mihaela ZUGRAVU[1], Muguraș CONSTANTIN[1], Aurora DOBRIN[1], Maria PARASCHIV[3, 4], Liliana BĂDULESCU[1]

[1]University of Agronomic Sciences and Veterinary Medicine of Bucharest, 59, Mărăști Blvd., District 1, Bucharest, Romania
[2]IMT Atlantique, 2 Rue Alfred Kastler, 44300 Nantes, France
[3]National Institute of R&D for Biological Sciences, 296, Spl. Independentei, District 6, Bucharest, Romania
[4]Research Center for Advanced Materials, Products and Processes - University Politehnica of Bucharest, 313, Spl. Independenței, District 6, Romania

Corresponding author email: mariaparaschiv@gmail.com

Abstract

The present study is part of a comprehensive study dedicated to the cultivation of halophytes species on salt affected soils, aiming on new value chains development from obtained biomass. The work is conducted on the monitoring the degree of soil purification, the biomass production and seeds yield, and their chemical composition. In this context, the paper contains information related to the oil content of Portulaca sativa sp. seeds, in order to produce 2nd generation biofuels. Extraction of oil from seeds via traditional method (Soxhlet method) and accelerated solvent extraction (ASE) were carried out. ASE method was applied because requires small quantities of solvent, sample and operating time. ASE appear to be the most suitable method and the optimal conditions were: pressure - 10.34 MPa, temperature - 105°C, residence time - 10 min, solvent - petroleum ether, extraction ratio of 1:40, dynamic extraction time - 30 min, and 0.3 g diatomaceous earth. The highest oil recovery achieved was 33.4%.

Key words: *accelerated solvent extraction, extraction parameters, Portulaca sativa.*

INTRODUCTION

Global climate change, water scarcity, and availability of nutrients in the soil (Guyer et al., 2018) are the main problems affecting all-natural resources availability (Borsai et al., 2018). Simultaneously, the population of the globe and its needs are constantly increasing, while resources are becoming less and less. Therefore, a sustainable approach is needed, targeting both need and consumption.

One of the sectors that needs attention, other than the main one for food, is energy. Energy demand is increasing along with population growth, urbanization, and living standards. As is known, the main categories of fuels are petroleum, natural gas, coal, natural gas, biofuels, nuclear energy, hydro energy, and other sources like solar, wind, geothermal. Of this, fossil fuels remain the main source of energy (Khan et al., 2014). The use of vegetable oils (palm, soya bean, sunflower, peanut, and olive oil), as fuel for engines dates long ago, and is promoted again. It is a renewable source and the ideal solution for global energy demands (Shereena & Thangaraj, 2009).

For now, the annual worldwide oil production is close to 135 Mt including palm, soybean, and rapeseed oils (Jolivet et al., 2013).

One of the answers to the hypotheses presented above is represented by halophyte plants. Using halophytes as crops would lead to reduce water consumption in agriculture and can be ideal for areas with water availability (Koyro et al., 2011). Some halophyte species (grasses, shrubs, and trees, etc.) can remove the salt from salt-affected soils through salt excluding, excreting, or accumulating (Hasanuzzaman et al., 2014; Karakaş et al., 2017).

Seeds of many halophytes (*Portulaca* sp., *Salicornia bigelovii*, etc.) contain appreciable quantities of oil and could be used as source of energy. Biofuels obtained using halophyte biomass could be a feasible alternative to conventional ones. The main advantage of using halophytes is that they don't compete with food (in terms of agricultural soil and water resources). Other aspect is the low impact on environment (Hameed & Khan, 2011). Non-edible biodiesel crops are expected to use lands that are largely unproductive and those that are located in poverty areas (Ahmia et al., 2014).

Portulaca sativa (Family Portulacaceae) is a possible candidate for this purpose. Is an annual plant widespread both in temperate and tropical regions. *Portulaca* is a grassy plant with freshly stems, succulent leaves, yellow or white small flowers and small black seeds (Rahimi et al., 2019).

Besides its pharmacological potential (Al-Shedd et al., 2015), *Portulaca* is highly adaptable to dry and saline conditions, being a strong candidate for areas with dry conditions and salty soils (Yazici et al., 2007).

Seeds are brown to black, oval, and tiny. A single plant may produce 240.000 seeds, and can germinate even after 5 - 40 years (Okafor et al., 2014).

Regarding the chemical composition of the oil from the seeds, related to the likelihood of obtaining biodiesel, the specialized literature is scarce. Thus, the main components found in *P. oleracea* are fatty acid content of the oil which was found to be composed of unsaturated (79%) and saturated (20.7%) compounds (Sodeifian et al., 2018).

MATERIALS AND METHODS

For this study were used two extraction methods: Soxhlet technique and accelerated solvent extraction.

The most popular solid-liquid extraction technique is called Soxhlet technique, which is practically carried out in an extraction apparatus of the same name (Figure 1). Soxhlet extraction is usually applied to solid or ″semi-solid″ samples. The samples to be analysed are first crushed and brought into a very fine powder form to enlarge the surface of contact in the extraction process. This method was used for various purposes, like vegetable oils (Dutta et al., 2011), flavonoids (Kaleem & Ahmad, 2018), anionic surfactant removing (Rowe, 2010), foods (Hammond, 2003), phenols (Luque de Castro & Priego Capote, 2010) etc. This method is based on a large difference between the boiling points of the solvent and those of the extracted analytes.

Figure 1. Soxhlet extraction system

Based on this property the extract is brought to a boiling temperature of the solvent, which will condense into a refrigerant and return to cartridge containing sample extract. By performing several extraction cycles, the efficiency of the process can be controlled so that the extraction efficiency is maximum.

However, two main drawbacks can occur in the Soxhlet extraction technique. The first refers to the fact that the extract is exposed throughout the process to the boiling temperature of the solvent, which, if very high, may affect some analytes in the sample, thermally labile. The second disadvantage is the low concentration in the end, due to the large amount of solvent used.

Accelerated solvent extraction (ASE, Figure 2) is an extraction method based on the use of high temperature and pressure to accelerate the dissolution kinetics and to break the analytical-matrix interaction bonds. For this reason, this method is also called liquid pressurized (solvent) extraction. In addition, increasing the temperature reduces the viscosity of the solvent, which makes it easier to penetrate the solid matrix of the sample. In this way, the extraction time is reduced from tens of minutes to a maximum of several minutes, and the extraction samples can be in small quantities. This type of extraction is recommended by the U.S. EPA for the extraction of solid samples from the environment like dioxins and furans, herbicides (Guzzella & Pozzoni, 1998), Organochlorinated Pesticides (OCPs), Organophosphorus Pesticides (OPPs), Persistent Organic Pollutants (POPs), (Hubert et al., 2001), Polybrominated Diphenyl Ethers (PBDEs), Polychlorinated Biphenyls (PCBs) (Brandli et al., 2006), etc. Also, the method is applied in fats and food safety domains (Abdul Mottaleb & Sarker, 2012).

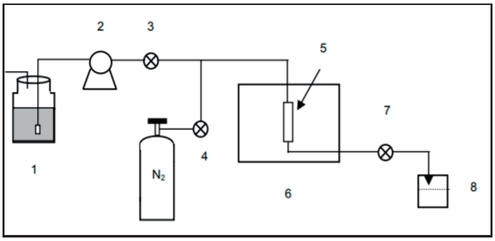

Figure 2. Components of an ASE system: 1 - the tank; 2 - pump; 3 - pump valve; 4 - purge valve; 5 - the extraction cell; 6 - thermostat enclosure; 7 - static valve; 8 - collection vial

The seed sample was minced to 1-2 mm. For accelerated solvent extraction method, the sample was mixed with diatomaceous earth. The sample: diatomaceous earth ratio used was 4: 1. ASE 350 model equipment was used (Figure 3).

Figure 3. ASE 350 system and extraction cell

For Soxhlet method, the sample: extraction solvent ratio was 1: 40. Extraction variants for accelerated solvent extraction (ASE 350) are presented in Table 1. In static ASE, the sample was extracted with solvent at elevated temperature and pressure conditions without any outflow of solvent (Mandal et al., 2015).

Table 1. ASE and Soxhlet extraction variants

Extraction solvent	Petroleum ether 100%	Petroleum ether 100%	n-Hexane	n-Hexane
ASE 350				
Temperature	105°C	105°C	80°C	80°C
Static time	10 min	10 min	10 min	10 min
No. of cycles	3	6	3	6
Washing volume	100%	100%	60%	60%
Purge time	60 sec	60 sec	60 sec	60 sec
Whole seeds	1.2 g	-	-	-
Sample 1	1.2 g	-	-	-
Sample 2	5 g	5 g	5 g	5 g
Soxhlet				
Sample 3	10 g	20 refluxes	15 min/reflux	

The advantage of the Soxhlet system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled (Nafiu et al., 2017).

RESULTS AND DISCUSSIONS

The factors which influence the oil extraction yield were: solvent of extraction, temperature, extraction time and liquid/solid ratio. Thus, optimal conditions for oil seeds extraction were obtained as follows: temperature 105°C, static time 10 min, and the number of cycles 3. Under these conditions the best value obtained for the oil extraction yield was 33.39 ± 0.013%. The obtained results are similar with those found by Petropoulos et al. (2020), 33.7% and 38.1%, respectively. Compared to ground seeds, the use of whole seeds, did not give positive results. The results obtained according to the extraction variants are presented in Table 2.

Table 2. Oil content in *Portulaca* sp. seeds

Variants	g oil/ sample	g oil %
Whole seeds	0	0
1.2 g shredded seeds, petroleum ether, 3 cycles	0.4008	33.4±0.013
1.2 g shredded seeds, n-hexane, 3 cycles	0.0994	8.25± 0.083
5 g shredded seeds, petroleum ether, 3 cycles	0.423	8.46±0.416
5 g shredded seeds, petroleum ether, 6 cycles	0.4586	9.17±0.383
5 g shredded seeds, n-hexane, 3 cycles	0.4214	8.43±0.378
5 g shredded seeds, n-hexane, 6 cycles	0.4994	9.98± 0.035
10 g shredded seeds, petroleum ether	1.671	16.71±0.273

Regarding Soxhlet extraction, the amount of oil obtained from the sample was 1.67 g. According to the obtained results found by Hoseini et al. (2019), it can be concluded that *Portulaca* weed seeds are a suitable source for biodiesel production.
Regarding the extraction solvent, the best variant was the one in which petroleum ether was used. The results are similar with those found by Uoonlue and Muangrat (2019) on *Camellia sinensis*.
Regarding the use of a larger quantity of sample 5 g and even if was kept the same number of cycles and the same solvent it was found that the amount of oil obtained was smaller than that obtained in the initial version of 1.2 g.
Among the experiment, the most influential parameter was solvent type. Although solvent sample ratio is reported to have no significant effect, this aspect has been shown to be partially demonstrated, the amount of solvent can be greatly reduced. Taking as reference other experiments on other types of samples, each optimized method is unique to the plants. However, regarding purity, advanced extraction technology such as ASE should be considered.

CONCLUSIONS

Comparing the two extraction methods used, we concluded that the oil content was higher for ASE method than Soxhlet (33.4% vs 16.71%). The extraction ratio was the same 1: 40. The differences between the 2 methods were related to the extraction time, the volume of the solvent and the amount of sample used. Thus, for the Soxhlet vs ASE method the extraction time was 5 h-1 h, the volume of the solvent 400 ml-100 ml, and the sample amount was 10 g-1.2 g.

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