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Optimization of Hemp Seeds(Canapa Sativa L.) Oil Mechanical Extraction

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The interest in hemp (Canapa Sativa L.) in the last decade is greatly increased for its multiplicity of uses: food; textile industry; cosmetics and biochemical. In addition, hemp seed oil is a great source of high quality nutrients and has multi-purpose natural remedies and It could be used as edible oil containing about 80% of essential fatty acids (excellent ratio of omega-6 to omega-3). The aim of this study was the optimization of hemp seeds oil mechanical extraction parameters in order to have a higher oil yield. The hemp variety was Futura 75, cultivated in Castelvolturno (CE), Southern Italy. Harvest has been carried out in September 2016 with a mechanical harvester. The test has been carried out using a mechanical screw press, powered by 2.2 kW electric motor, using an 8 mm nozzle. The variable parameters were: temperature of extraction (50 °C and 70 °C), measured with a temperature sensor placed on press head; two rotational screw speeds (22 rpm and 32 rpm) and seeds pre-treatment (heated at 50 °C for 1h in a laboratory oven and not heated). The processed samples weighted 1000 g each. After the extraction, oil was centrifuged at 3500 rpm for 20 minutes with an laboratory centrifuge to determine the yield of pure oil as percentage of extracted oil (g) on the total oil extractable basis. During the tests, the performances of the press were monitored: initial and final extraction temperatures have been recorded with a RTD (Pt100) temperature sensor; extraction time was recorded with a laboratory stopwatch. Results show that the combination of high extraction temperature and slow rotational screw speed has a positive effect on oil yield as well as seeds pre-treatment. Further studies are scheduled in order to evaluate the effect of extraction parameters on oil quality in terms of acidity, sensory profile, volatile compounds and fatty acids composition.

1. Introduction

Hemp is considered one of the first cultivated crops. Sources estimate hemp was first cultivated between 4000 and 6000 years ago, in China (Randall Fortenbery and Bennett, 2004). Traditionally, hemp was used for fibre and oil production. Cannabis Sativa L. is an annual herbaceous crop, usually dioecious. For many years, this crops has been a traditional crop in Italy, but at present it almost disappeared from Italian land due to problems with cannabinoid content in plants and the negative attitude of Italian Administration towards this crop. The Cannabis Sativa L. was prohibited in Italy since 1980 (Cappelletto et al., 2001). Since 1996, hemp varieties containing less than 0.2 % THC can be grown (Aladic et al., 2014). In addition to cultivation finalized to fibre production, hemp has been cultivated also for food production. Hemp seeds and the products derived from them like edible oil, in addition to their nutritional value, have shown positive effects on haematic cholesterol, triglycerides, blood pressure, dermatitis care. Also, hemp seed oil could be used as integrator for traditional medicine preparation (Rovellini et al., 2013). Hemp oil seed extraction is usually carried out by mechanical cold-pressing methods in order to preserve oil quality (Da Porto et al., 2011). Mechanical press extraction, respect to solvent extraction techniques, seems to be more acceptable for health, safety, economic and environmental reasons (Yusuf et al. 2014). The aim of this study was to investigate the effect of the combination of extraction parameters, such as extraction temperature, seeds preheating and screw speed rotation, on oil yield in order to create an extraction protocol that creates optimal extraction conditions.

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2. Materials and Methods

2.1 Hemp seed material

In this study, hemp seeds were harvested from experimental fields in Castel Volturno (CE) in Southern Italy. Seeds were cleaned to remove impurities with a pneumatic separator, then stored in a warehouse at temperature between 15 °C and 24 °C (Faugno et al., 2016; Galloway, 1976; Karaj and Muller, 2011). Sample seed moisture content was kept constant to 9 % on wet basis. Moisture seed content was determined by standard hot-air oven (Binder, BD115, Germany) method at 105 ± 1 °C to constant weight (Faugno et al., 2016; Garnayak et al., 2008). The oil content of untreated samples was 33.77 ± 0.12 % (w/w) and it was determined by Accelerated Solvent Extraction utilizing an ASE®200 System Dionex.

2.2 Mechanical oil extraction procedure

Hemp oil was extracted from seeds with a mechanical screw press (Figure 1), powered by 2.2 kW electric motor. Different sensors were installed for measuring temperature (RTDs, Pt100) in two different positions (T1 – T2 in Figure 1).

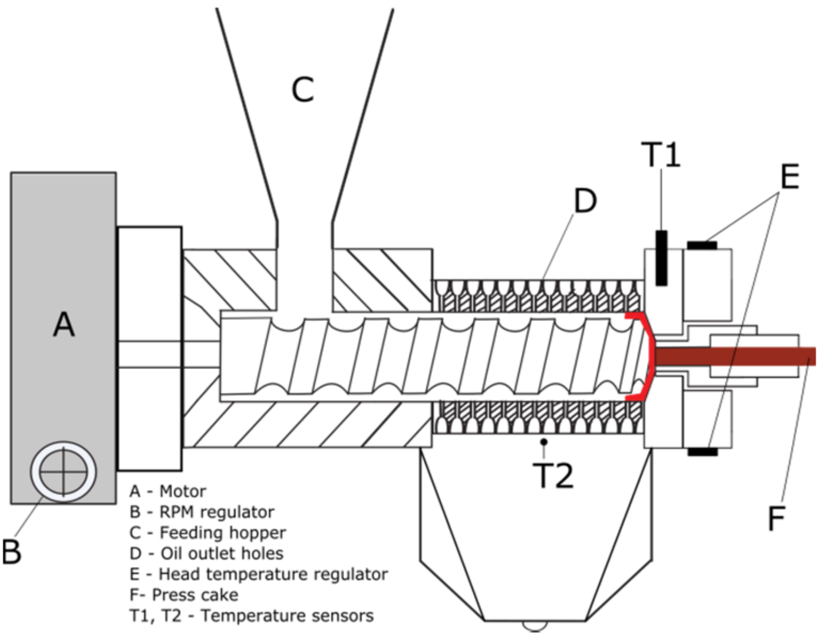

Figure 1: Mechanical screw press for tobacco oil extraction and installed sensors used in this study

Screw speed was adjusted by a variable speed regulator (mechanical control) and it was measured with a rev counter (Faugno et al., 2016; Karaj and Muller, 2011). The sensors were connected to a data-logger (National Instruments cDAQ-9171) and data transferred via data-logging software (National Instruments LabVIEW 2014) to a laboratory computer. The pure oil yields were calculated with the methodology used by Faugno et al. (2016). The extraction conditions were evaluated because they caused a variation of oil yield (Kraljic et al., 2013; Faugno et al., 2016).
In order to evaluate the effect of mechanical extraction parameters on hemp oil yield, the following conditions were considered:

1) Two different initial temperatures of extraction: 50 °C (T50) and 70 °C (T70) of head press (Faugno et al. 2016; Tambunan et al. 2012);
2) Thermal pretreatment of seeds: yes (H) or not (NH). The seeds were dried for 1 h at 50 °C and then processed (Faugno et al. 2016; Galloway 1976; Singh et al. 2000);
3) Two different rotational speeds of the screw: 22 rpm (ω22) and 32 rpm (ω32) (Faugno et al. 2016; Karaj and Muller 2011).

In this study, two different experimental setups were carried out with combinations of three different parameters, utilizing an 8 mm nozzle size (Faugno et al. 2016). In Setup 1 the temperature of the press was kept constant at 50 °C, whereas the pretreatment was varied in combination with screw speed.

In Setup 2, the temperature was constantly at 70 °C and the combinations between screw speed and pretreatment were the same as Setup 1 (Table 1).

Table 1: Experimental setup: 8 mm nozzle size; T50 and T70 temperatures of extraction (°C); H and NH thermal pre-treatment (yes or not); ω32 and ω22 rotational speeds of screw (rpm)

Setup 1	Setup 2
T50_H_ω32	T70_H_ω32
T50_H_ω22	T70_H_ω22
T50_NH_ω32	T70_NH_ω32
T50_NH_ω22	T70_NH_ω22

For each sample, 1000 g of seeds were used and three replications were done (Faugno et al. 2016; Unquiche et al. 2008). The extracted TSO was collected and stored in light-proof glass containers and centrifuged in a laboratory centrifuge (ALC T535 PK130R) at 3500 rpm for 20 minutes in order to remove components that settled during storage (Unquiche et al. 2008). The solid by-product (Hemp seed cake) was stored in vacuum packages (Faugno et al. 2016; Unquiche et al. 2008), at 4°C in the dark. Oil yield was calculated as percentage of extracted oil on the total oil extractable basis. The extractable oil basis has been calculated as sum of extracted oil and oil content in press cake. The following Eq(1) has been used in this study (Faugno et al., 2016; Unquiche et al., 2008; Karaj and Muller, 2011)

$$O_y = \left[\frac{O_e}{O_e + O_c}\right] * 100 \tag{1}$$

with O_y as oil extraction yield in % (w/w); O_e as extracted oil in g and O_c as oil content in press cake in g as described in the “Determination of oil content in press cake” section. In this study, the throughput has been calculated as ratio between the seed sample weight (kg) and the time (h) required for pressing the sample.

2.3 Determination of oil content in press cake

The crude oil and press cake were weighed after each experiment and taken to the laboratory for additional analysis. The percentage of oil content in press cake was determined with ASE®200 (Accelerated Solvent Extraction) Dionex, in the following conditions using hexane as a solvent:

- 4 g of tobacco press cake were ground in a Retsch MM400 mixer mill for 2 min at 25 Hz, in presence of diatomaceous earth (Dionex ASE Prep DE), as a dispersant and drying agent, in a 1:1 w/w ratio (Modestia et al., 2013);
- oven temperature set at 105 °C;
- pressure about 1500 psi, static time 10 min, rinse volume 100 %, purge time 90 s and 3 static cycles.

After the extraction, the vials (previously weighted) containing the extract were placed in a TurboVap II (Zymark Corporation, Horsham, PA, USA) to evaporate the solvent using dry nitrogen. The oil yield was measured by weighting and expressed as percentage on dry weight basis. Seed moisture content was determined after drying seed samples for 3 h at 105 °C. The procedure accuracy was evaluated by determining oil yield of IRMM certified reference material CRM BCR-447 (Medium Oil Content Rapeseed).

2.4 Statistical analysis

Data were analyzed using Analysis of Variance (ANOVA) with temperatures, thermal pretreatment and rotational speeds of the screw as factors. Statistical significance of the differences observed among mean values was assessed using Tukey multiple comparisons of means. A probability of $p \leq 0.05$ was considered significant. The data has been elaborated with OLS (Ordinary Least Squares) regression following the model:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 \tag{2}$$

The y represents the dependent variable that is the phenomenon explained, the x represents all the factors used to explain the phenomenon, and the β represents the magnitude associated to each independent variable. According to Anania et al. (1995) data has been normalized in order to compare the results. The R Studio statistical software (version 3.1.2) was used to analyze the collected data.

3. Results and discussion

Oil yield from hemp seeds samples at different processing conditions is shown in Table 2. The highest oil recovery of 73.38 ± 0.307 % (w/w) was achieved by using Setup 2 (T70_H_ω22) and the lowest oil recovery efficiency of about 58.40 ± 0.49 % (w/w) by using Setup 1 (T50_NH_ω32). Oil residue in press cake was higher in Setup 1 (T50_NH_ω32) and it was lower in Setup 2 (T70_H_ω22).

Table 2: Results of mechanical extraction. O_y = oil yield expressed as % (w/w); O_c = oil residue expressed as % (w/w); TP = throughput as kg/h

		O_y	O_c	TP
Setup 1	T50_H_ω32	64.77 ± 0.65	12.4 ± 0.47	19.7 ± 0.19
	T50_H_ω22	70.13 ± 0.62	10.7 ± 0.42	13.1 ± 0.29
	T50_NH_ω32	58.40 ± 0.49	13.9 ± 0.55	19.5 ± 0.30
	T50_NH_ω22	68.43 ± 0.68	10.9 ± 0.65	14.1 ± 0.06
Setup 2	T70_H_ω32	68.93 ± 0.94	11.9 ± 0.70	18.8 ± 0.60
	T70_H_ω22	73.38 ± 0.30	10.4 ± 0.26	12.9 ± 0.62
	T70_NH_ω32	61.52 ± 0.28	13.2 ± 0.18	20.0 ± 0.56
	T70_NH_ω22	67.08 ± 0.45	11.9 ± 0.09	14.3 ± 0.11

Table 2 shows the average throughput of 20.0 ± 0.56 (kg/h) calculated for high rotational screw speed and an average throughput of 12.9 ± 0.62 (kg/h) calculated for slow rotational screw speed. According to Karaj and Muller (2011), Tambunan et al. (2012) and Faugno et al. (2016), higher oil yield has been obtained with seed preheating, high press temperature and slow rotational screw speed. The results of Tukey multiple comparison of means (Tukey HSD) analysis on the experimental data is shown in Figure 2.

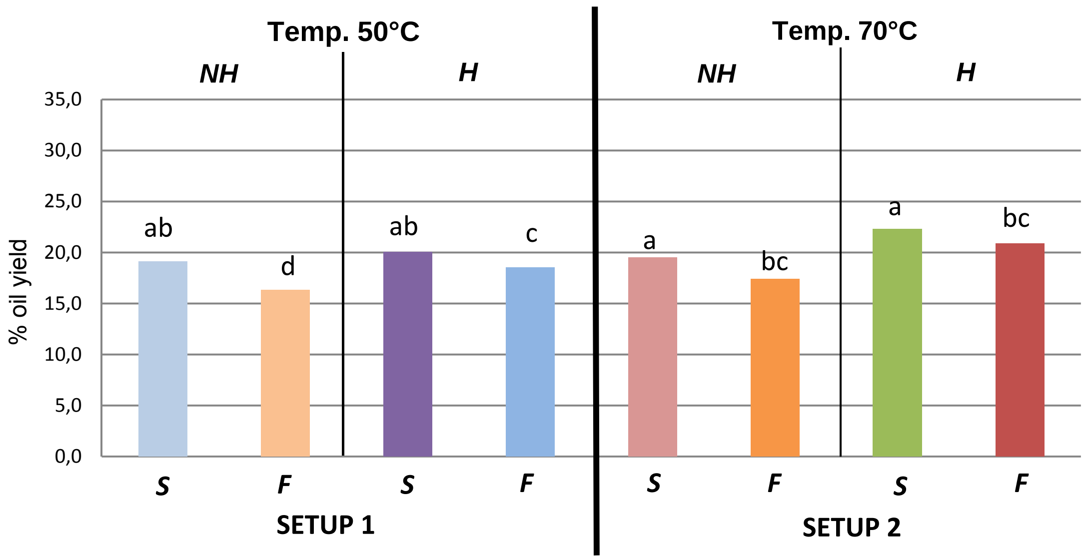

Figure 2: Results of Tukey HSD analysis on oil yield

The results show that the extraction temperature, the preheating and the speed rotation of screw respectively gave significant effect to the oil yield. In Setup 1 it is possible to notice a significant difference ($p < 0.05$) between preheated samples and not preheated samples only using an high rotational screw speed. Moreover, the rotational screw speed factor determines significant differences ($p < 0.05$) between slow and fast samples both in preheated and not preheated seeds conditions. In Setup 2, as in Setup 1, the rotational screw speed has a significant influence ($p < 0.05$) on oil yield both in preheated seeds and not preheated seeds conditions. Comparing Setup 1 and Setup 2 in not preheated seeds conditions and only using an high rotational screw speed, it is possible to notice a significant difference ($p < 0.05$) in oil yield. The results show that the rotational screw speed determines a significant difference in oil yield ($p < 0.05$) among slow and fast samples in both setups.

All the combinations in this study, setting the slow rotational screw speed, show no significant differences in oil yield. Linear regression standardized coefficients are reported in Table 3. Coefficient β_1 associated to extraction temperature predictor, is significant and positive. Accordingly, with a temperature rise there is an increase of oil yield. Coefficient β_2 associated to pretreatment predictor, is significant. Coefficient β_3, associated to rotational screw speed predictor, is significant and positive. This leads to an increasing of oil yield decreasing the rotational screw speed. The coefficient of determination (Multiple R-Squared) evaluates the effectiveness of the model that is the proportion of variability of y explained by the explanatory variables considered. In this study, R-Squared is equal to 0.91, meaning that 91 % of data is explained by the explanatory variables.

Table 3: OLS (Ordinary Least Squares) regression results

	Estimate value	t value	Pr(>|t|)	
(Intercept)	77.6758	220.130	< 2e-16	***
Temperature of extraction (β_1)	0.6417	1.818	0.064	*
Pretreatment (β_2)	-2.7317	-7.741	1.93e-07	***
Rotational speed of screw (β_3)	4.0050	11.350	3.61e-10	***

Significance codes: < 0.001 ‘***’ < 0.01 ‘**’ < 0.05 ‘*’

4. Conclusions

The aim of this study is to evaluate hemp oil extraction yield under different conditions: extraction temperature, seeds preheating and screw rotational speed, in order to create an extraction protocol with respect to oil recovery. According to the results, the oil yield is higher with an high extraction temperature, preheating the seeds and setting the lowest screw rotational speed.

With a combination of these three factors, results show a significant influence of extraction temperature and preheating on oil yield with an high screw rotational speed. Otherwise, with low screw rotational speed, extraction temperature and preheating has no significant influence on oil yield.

In this study, results showed that screw rotational speed has an higher influence on oil yield than extraction temperature and seeds preheating, as demonstrated by regression values β_3, β_2 and β_1.

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