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Optimization of water extraction of naturally emulsified oil from maize germ

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ABSTRACT

A design of experiments-based experimental strategy is proposed to develop an effective and rapid water extraction process for maximizing the yield of naturally-emulsified oil based on oil bodies from maize germ. Orthogonal array (D9) of Taguchi, the signal-to-noise ratio, the main effects, and the analysis of variance (ANOVA) were adopted to pre-test the process parameters (germ flour particle size (d_p), water-to-solid ratio (w), pH and extraction time (t)) for their effect on the extraction yield (Y) and the diameter of oil droplets ($d_{3,2}$). Water extraction of emulsified oil from germ flour of $d_p < 0.85$ mm at pH 9.0 was optimized using response surface methodology and central composite design (independent variables: w, t, and n for number of cycles). The quadratic model fitted well to Y. The extraction yield of emulsified oil of 99.30 ± 0.56% (w = 3:1 g/g, t = 14 min, n = 5) is the highest reported in the literature.

1. Introduction

Triglycerides in oil-rich plant sources, such as seeds and germs, are stored in the form of cell organelles called oil bodies (Tzen, Lie, & Huang, 1992). Following their extraction by aqueous media, oil bodies in the resulting dispersion appear to be spherical. In their natural cell environment they obtain a polygonal shape due to surface stresses developing as a result of intimate contact with other organelles (Nikiforidis, Kiosseoglou, & Scholten, 2013). However, in spite of this, oil bodies tend to retain their integrity and do not coalesce into larger ones due to the presence at their surface of an elastic, mixed protein-phospholipids membrane which endows them with considerable electrostatic and steric stability (Huang, 1994). In addition to their physical stability, the oil bodies are also highly stable against chemical stresses. The stability of their triglycerides against oxidation is attributed to the surface protein/phospholipids layer that surrounds the lipid core (Fisk, White, Lad, & Gray, 2008).

The remarkable physical stability of oil bodies may constitute a serious problem when vegetable oil extraction from crushed oil-rich materials by aqueous media, as an alternative way to avoid the use of the highly polluting and industrially hazardous organic solvents such as hexane, is applied (Chabrand, Kim, Zhang, Glatz, & Jung, 2008; Lamsal & Johnson, 2007). Their high stability, however, can be advantageous when the aim of aqueous extraction is not to break down the oil bodies' surface and bring about the liberation of the vegetable oil but instead recover intact oil bodies in the form of a natural oil-in-water emulsion (Iwanaga et al., 2007; White et al., 2008). The benefits of this approach are more than obvious since such an emulsion made up of small sized oil droplets that exhibit considerable stability against coalescence can be exploited in the preparation of novel food emulsion products (Chen, McClements, Gray, & Decker, 2012; Matsakidou, Biliaderis, & Kiosseoglou, 2013; Nikiforidis, Biliaderis, & Kiosseoglou, 2012; Nikiforidis & Kiosseoglou, 2009; Rosenthal, Pyle, & Niranjan, 1998; Yu, Li, Du, Zhang, & Xu, 2013).

Naturally emulsified oil extraction from a number of oil-rich raw materials with the use of aqueous means, often combined with enzyme treatment, has been the subject of several research papers which indicated that the extraction yield is determined by a number of parameters such as pH value, temperature and degree of crushing (Kapchie, Towa, Hauck, & Murphy, 2010; Kapchie, Wei, Hauck, & Murphy, 2008; Nikiforidis & Kiosseoglou, 2009; Rosenthal et al., 1998). Nikiforidis and Kiosseoglou (2009) reported that the main parameters which influence the yield of oil bodies' extraction from maize germ are the degree of raw material comminution, the pH value, and the extraction cycles applied. Other parameters which may also affect the extraction yield, such

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as the water-to-solid ratio and extraction time, were not considered by these investigators. In addition, a more thorough study of extraction parameters should also consider their interaction effects in maximizing the yield and thus minimizing operation cost and increasing process capacity.

Taguchi method and Response Surface Methodology (RSM) are the most commonly used Design of Experiments (DoE) techniques in optimization studies. The Taguchi method uses orthogonal arrays to screen a large number of controllable and noise factors and mark the significant ones and their levels. For continuous factors, this technique is not normally used for experimental optimization due to its inability to determine the best combination of factor values within the specified region of interest. This problem can be overcome by using RSM that allows for finding an approximation suitable to fit to the data from experiments at various points in the experimental space defined by Central Composite Design (CCD) and predict the optimum factor combination (Arteaga, Li-Chan, Vazquez-Arteaga, & Nakai, 1994). In most research based on DoE, either Taguchi method or RSM are applied. However, the combined use of the above approaches was proved to be useful in engineering optimization applications (Hou, Su, & Liu, 2007).

This study was aimed to integrate the two methods in order to optimize the most critical variables to maximize water extraction yield of oil bodies from maize germ while at the same time protecting them from coalescence. To achieve this aim, process parameters were pre-tested using the Taguchi method, followed by prediction of optimum combination of the selected significant ones to get the desired response values using RSM and CCD.

2. Materials and methods

2.1. Water extraction of naturally emulsified oil

Maize germ was kindly provided by a local mill (Karanika, Alexandreia, Greece) as a by-product and subjected to milling (Braun ZK100, Kronberg im Taunus, Germany) for 30 s. The resulting flour was separated into three fractions by employing sieves of a mesh size of 0.85 and 2 mm. Part of each fraction with desired particle size (d_p) value was initially soaked in appropriate amount of deionized water to reach the desired water-to-solid ratio (w) value, at 25 °C for 2 h. During this time period, the mixture (total volume of 450 mL) was continuously stirred with the aid of a mechanical stirrer (Kika Labortechnik, Staufen, Germany) while the pH was kept constant, at the desired value (pH), by adding NaOH or HCl (Sigma Chemical, St. Louis, USA) solution, accordingly. Following storage at 4 °C for 12 h to soften and loosen the germ structure, the mixture was subjected to intensive agitation with a four blade blender (Braun ZK100, Kronberg im Taunus, Germany) for oil body extraction. At the end of the target extraction time (t), the resulting dispersion was filtered through three layers of cheesecloth and the extracted germ residue was used to determine the remaining oil. The filtrate was centrifuged at 1700 *g* for 15 min (Filarbo SV11, Meyzieu, France) and the supernatant was used for determining oil bodies mean diameter (see Section 2.3). To apply additional extraction cycles (n), the germ residue from the single extraction cycle was then mixed again with appropriate amount of deionized water, and after pH adjustment the mixture was subjected again to intensive agitation and filtered through the cheesecloth. The oil body extraction procedure was repeated up to 4 more times.

Aqueous extraction yield of naturally emulsified oil was calculated indirectly by determining the oil content of the extracted germ residue at the end of the extraction steps and subtracted from the initial oil content of the raw germ, according to Eq. (1):

$$Y = 100 \cdot \left(1 - \frac{m}{m_0}\right) \quad (1)$$

where

Y: extraction yield of emulsified oil, %
m: oil content of extracted germ residue, (% dry matter)
m_0: oil content of raw germ, (% dry matter)

2.2. Moisture and oil determination

The determination of moisture (%, w/w) and of m and m_0 was conducted by the gravimetric method and Soxhlet extraction method with petroleum ether, respectively, according to AOAC (Association of Official Analytical Chemists, 1992).

2.3. Measurement of droplet diameter distribution

Droplet diameter distribution of oil body-based dispersion was determined with the aid of a laser light scattering instrument (Malvern Mastersizer 2000, Malvern, UK) (Nikiforidis & Kiosseoglou, 2009). Measurements are reported as the surface weighted ($d_{3,2} = \sum n_i d_i^3 / \sum n_i d_i^2$) mean diameter, where n_i is the number of droplets with a diameter of d_i. Sodium dodecyl sulfate (SDS) (Sigma Chemical, St. Louis, USA) at a concentration of 0.01% (w/w) was added to ensure the full dispersion of any aggregated droplets.

2.4. DoE for selection of extraction parameters: Taguchi method

The input process parameters, (d_p, w, pH, t) were pre-tested for their effect on Y and oil droplet size ($d_{3,2}$). In the screening study, Taguchi orthogonal array experimental design of four factors at three levels (L9) (3^4) (Table 1) was employed using R language and environment for statistical computing (version 2.15.2). Statistical analysis of the experimental results was expressed in terms of the mean values to determine the main effect for each factor and its corresponding level and the signal-to noise (S/N) ratio to recognize

Table 1
Taguchi orthogonal array experimental design of four factors at three levels and the observed responses for emulsified oil extraction yield (Y) and oil droplet diameter ($d_{3,2}$).

Parameter	Variable	Level 1	Level 2	Level 3
Germ flour particle size (μm)	d_p	<0.85	0.85–2	>2
Water-to-solid ratio (g/g)	w	3:1	5:1	10:1
pH	pH	3.0	7.0	10.0
Extraction time (min)	t	0.17	0.5	1

Trial	Independent variables				Response variables[a]	
	Germ flour particle size (μm)	Water-to-solid ratio (g/g)	pH	Extraction time (min)	Yield (%)	Oil droplet diameter (μm)
	d_p	w	pH	t	Y	$d_{3,2}$
1	<0.85	3:1	3.0	0.17	0.00 ± 1.17	0.269 ± 0.036
2	<0.85	5:1	7.0	0.5	24.26 ± 4.59	0.290 ± 0.087
3	<0.85	10:1	10.0	1	61.66 ± 4.53	0.157 ± 0.023
4	0.85–2	3:1	7.0	1	16.30 ± 0.47	0.594 ± 0.082
5	0.85–2	5:1	10.0	0.17	0.00 ± 1.05	0.162 ± 0.036
6	0.85–2	10:1	3.0	0.5	0.00 ± 1.10	0.200 ± 0.019
7	>2	3:1	10.0	0.5	23.88 ± 0.59	0.201 ± 0.016
8	>2	5:1	3.0	1	0.00 ± 1.22	0.254 ± 0.037
9	>2	10:1	7.0	0.17	9.03 ± 0.66	0.284 ± 0.049

[a] Data are presented as average ± standard deviation ($n = 3$).