1. ABOUT THE DATASET

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Title: Data used in the article ‘Optimisation of the physicochemical stability of extra virgin olive oil-in-water nanoemulsion: processing parameters and stabiliser type’

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Organisations: University of Reading

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Description: This dataset contains data obtained from experimental work on the characterisation of the interfacial properties of emulsifiers (Tween 20 and soy lecithin), the influence of homogenisation processing conditions (pressure and number of cycles) on physicochemical properties and stability of emulsions. The data was obtained using a high-speed homogenizer, a high-pressure homogenizer, a pendant drop analyser (interfacial tension), a spectrophotometer, a rheometer (viscoelastic behaviour) and a dynamic light scattering (DLS) instrument (mean droplet diameter and polydispersity index).

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Jansuda Kampa, Anastasios Koidis, Sameer Khalil Ghawi, Richard Frazier and Julia Rodriguez-Garcia. Optimisation of the physicochemical stability of extra virgin olive oil-in-water nanoemulsion: processing parameters and stabiliser type. 2022. Food and Bioprocess Technology. In preparation.

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2. TERMS OF USE

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3. PROJECT AND FUNDING INFORMATION

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No funding was received.

4. CONTENTS

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Data processing and preparation activities

Data was collected in Excel files. Different tabs have been assigned for different measurements. For data presentation an index tab at the beginning of each Excel file was created with the sample nomenclature, an explanation of the content of the file and a description of each of the variables studied. Data replicates are presented in columns with the heading ‘Rep’ or ‘Rep X’ (e.g. Rep1, Rep2, Rep 3).

File listing

1. ‘EmulsifiersCharacterisation’: this file contains data of the interfacial tension and rheological analyses done in the emulsifiers including Tween 20 and soy lecithin in terms of:
   1. Table 3. Characterisation of interfacial tension
   2. Figure 1. Storage modulus and loss modulus of Tween 20 and soy lecithin solutions
2. ‘PressureOnEmulsionProperties’: this file contains data on the physical and chemical properties of nano/emulsions formulated with Tween 20 (5%) or soy lecithin (5%):
   1. Table 1: Effect of homogenisation pressure on mean droplet diameter
   2. Table 1: Effect of homogenisation pressure on polydispersity index
   3. Table 1: Effect of homogenisation pressure on lipid oxidation
3. ‘PressureCyclesOnNanoemulsionProperties’: this file contains the data of the analyses to evaluate the effect of HPH pressure and number of cycles on the physical and chemical properties of nano/emulsions stabilised with Tween 20 and lecithin:
   1. Figure 2 (A, B): interactions between the pressure and number of cycles for the MDD of nanoemulsions formulated with Tween 20 and lecithin, respectively.
   2. Figure 2 (C, D): interactions between the pressure and number of cycles for the PDI of nanoemulsions formulated with Tween 20 and lecithin, respectively.
   3. Figure 2 (E, F): interactions between the pressure and number of cycles for the thermal stability of nanoemulsions formulated with Tween 20 and lecithin, respectively. Different capital or lower-case letters above bars indicate significant differences between samples (p < 0.05)
4. PropertiesOfNanoemulsions: this file contains data of the analysis done to assess the effect of surfactant type on the physical and chemical properties of nanoemulsions:
   1. Table 2: Mean droplet diameter (MDD)
   2. Table 2: Polydispersity index (PDI)
   3. Table 2: Thermal stability (TS)
   4. Table 2: Thiobarbituric acid reactive substances (TBARS)

Variables explanation:

1. ‘EmulsifiersCharacterisation’:
   * Storage modulus: G'
   * Loss modulus: G"

5. METHODS

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Please see Materials and Methods section in the related article Kampa et al. 2022 (in preparation), which includes materials, nanoemulsion preparation, interfacial tension and rheological properties of emulsifiers, and physicochemical properties of emulsions (mean droplet diameter, polydispersity index, thermal stability, and thiobarbituric acid reactive substances).

**Materials**

Nanoemulsions were elaborated with extra virgin olive oil (EVOO) (14.26% of saturated fat, 77.69% of monosaturated fat and 8.04% of polyunsaturated fat (Napolina brand, UK retail market) and two types of emulsifiers: Tween 20 (Polyoxyethylene) with hydrophilic-lipophilic balance (HLB) value of 16.7 was used as synthetic nonionic surfactant (Sigma-Aldrich Co., Ltd, UK) or soy lecithin (HLB value ranges between 2-8) was used as zwitterionic surfactant, (Louis Francois Co, France). The molecular weight of soy lecithin and Tween 20 are 643.9 and 522.g/mol respectively (National Center for Biotechnology Information, 2021a, 2021b). Trichloroacetic acid, thiobarbituric acid, and malondialdehyde (MDA) were purchased from Sigma-Aldrich Co., Ltd (UK), and hydrochloric acid purchased from Fisher scientific Co., Ltd (UK). High purity water was used for the preparation and dilution of reagents.

**Nanoemulsion preparation**

The emulsion preparation procedure was based on the methods described by Arancibia et al. (2016) and Taha et al. (2018) with some modifications. Emulsions were prepared in three steps. Firstly, a magnetic stirrer (Model SS3H, ChemLab, UK) was used to prepare the aqueous phase dispersing Tween 20 and soy lecithin (5% w/w) in water (85% w/w) at 200 rpm for 30 min at ambient temperature to ensure complete dispersion. Then the oil was added (10% w/w) to the aqueous phase during continuous stirring. Secondly, the emulsions were homogenized with a high-speed homogenizer (Model L4RT, Silverson, Chesham, UK) at 10,000 rpm for 10 min. Thirdly, the coarse emulsions were passed through a high-pressure homogenizer (8.30H, Rannie APV, Denmark). For the evaluation of the effect of the homogenisation pressure on emulsion properties, the coarse emulsions were homogenised at 1, 200, 400, 600 and 800 bars for 1 cycle. For the study of the effect of cycle number on emulsion properties, the coarse emulsions were passed through the high- pressure homogeniser for 1, 2 and 3 cycles at different homogenisation pressures (200 and 400 bars). Then the emulsions were left to cool down for 2 hours at ambient temperature before measurements. All emulsions were prepared in triplicates.

**Interfacial tension and critical micelle concentration of surfactants**

The interfacial tension was determined according to the method of Bai et al. (2016) and Luo et al. (2017). The interfacial tension between oil-water interface, with different emulsifiers (Tween 20 at 5% w/w or soy lecithin at 5% w/w) was determined using a pendant drop analyser (DS4270, Krüss GmbH, Hamburg, Germany) at 20 °C. An axisymmetric drop (20 µL) of surfactant dispersion was delivered and allowed to stand at the tip of the needle inside a quartz container of extra virgin olive oil (9 mL) for 15 min, to achieve emulsifier adsorption at oil-water interface. Three analytical repetitions of each measurement were done for each emulsion batch.

The critical micelle concentration (CMC) of each emulsifier was determined according to the method of Mukherjee et al. (2013) and El-Sukkary et al. (2008). Several concentrations of Tween 20 and soy lecithin were prepared: 0.00, 0.0001, 0.001, 0.01, 0.1, 1.0, 5.0 and 10.0 %w/w. CMC was determined at the intersection points of the interfacial tension values versus the surfactant concentration (logarithm) plot. Three analytical repetitions of each measurement were done for each emulsion batch.

**Physical and chemical properties of nanoemulsions**

**Rheological properties of emulsions**

The viscoelastic behaviour of surfactant solutions was determined using a rheometer (Anton Paar MCR 302, Anton Paar, Graz, Austria) equipped a Peltier temperature control device. A serrated parallel plate geometry was used; the diameter of lower stationary plate (PPTD 200/56/1) and superior plate (PP50/ P2) was 50 mm and the gap between the plates was 1 mm. The samples were allowed to rest in the measurement position for 5 min for relaxation and temperature equilibration (20 °C). Strain sweeps were carried out at strain amplitude range of 0.001 to 1000 % at a constant frequency of 1 Hz in order to determine the linear viscoelasticity region (LVR). The LVR was identified where the storage modulus (G') and loss modulus (G") were not influenced by applied strain from this region, constant strain amplitude of 10% was selected. Frequency sweeps between 0.1 to 100 Hz were performed at constant stress amplitude (10%). The G' and G" moduli values were recorded; G' characterizes of the elastic nature or solid-like behaviour of a substance, while G" is indicative of viscous nature or liquid-like behaviour of a substance. Measurements were performed in duplicate in two emulsion batches.

**Measurement of emulsion mean droplet diameter (MDD) and polydispersity index (PDI)**

Particle size and polydispersity index of emulsions were determined in a dynamic light scattering (DLS) instrument (Zetasizer Nano ZS, Malvern Instruments Ltd., Worcestershire, UK) following the method of Guerra-Rosas et al. (2016) and Sharif et al. (2017). Emulsions were diluted 100-fold with deionized water and agitated to avoid multiple light scattering effects. The dispersion was decanted into polystyrene cuvettes for measuring MDD and PDI at wavelength of 633 nm at 25 °C. Three analytical repetitions of each measurement were done for each emulsion batch.

**Thermal stability (TS)**

Emulsion stability at high temperature was determined as described by (Sahafi et al., 2018). Each emulsion (10 mL) was heated in a water bath at 80oC for 30 min followed by centrifugation at 1200 g for 10 min. The height (mm) of initial emulsion, cream layer and sedimentation phase were measured with a Digital Vernier Caliper. Emulsion thermal stability was calculated according to the Eq. 1:

where was the height of initial emulsion (mm), was the height of sedimentation phase (mm) and was the height of cream layer (mm).

**Determination of thiobarbituric acid reactive substances (TBARS)**

TBARS were determined according to the method of (Qiu et al., 2015; Sharif et al., 2017) with some modifications. Briefly, 1 ml of the emulsions was added to 5 ml of thiobarbituric acid (TBA) solution, which was prepared by mixing 15 g of trichloroacetic acid (TCA), 0.375 g of TBA and 2.1 g hydrochloric acid (37% w/w). Samples were heated in a water bath at 95 °C for 10 min, then the samples were allowed to cool down to room temperature for 10 mins, followed by centrifugation (Heraeus Multifuge 3SR Plus Centrifuge, Thermo Scientific Ltd., UK) at 10,000 g for 15 mins. The absorbance of the supernatant was measured at 532 nm using a UV spectrophotometer (CECIL CE 1021 1000 Series, Cecil Instruments Ltd., UK). The absorbance of the samples was measured against a blank solution (7.5% w/v TCA). The concentrations of TBARS values were determined by using a standard curve prepared using malondialdehyde MDA standard (4.17 M). A concentration of MDA standards between 0.02 to 0.10 mM were prepared where linear response was observed coefficient correlation (R2) = 0.9987. Three analytical repetitions of each measurement were done for each emulsion batch.

**Statistical analysis**

Statistical analysis of the data was performed using IBM SPSS 25 (Armonk, NY: IBM Corp, USA). To assess the effect of the pressure (1, 200, 400, 600 and 800 bars) on the properties of nanoemulsions one-way analysis of variance (ANOVA) and Tukey’s HSD test were used to evaluate the mean values’ difference (*p* < 0.05). Then, a two-way ANOVA was conducted to assess the effect of the processing conditions: pressure (200 bars and 400 bars) and number of cycles (1, 2, 3 cycles)). Furthermore, a two-tail paired t-test was used to compare the effect of the two-surfactant studied (tween 20 and soy lecithin) on emulsion properties.The experimental data was analysed and reported as means and standard deviations.

The regression ANOVA was applied to determine the best HPH conditions to produce stable nanoemulsions. This regression ANOVA was employed using the Statistical Analysis System (SAS) software (SAS Institute Inc., USA). The regression model of the influence of high-pressure homogenisation processing parameters were analysed; homogenisation pressure at 1, 200, 400, 600 and 800 bars and the cycle number of 1, 2 and 3 were optimized in order to minimize the response variables under study of MDD, PDI, thermal stability (TS) and TBARS, and to maximize the response variables under study of TS using response surface regression. The empirical second-order polynomial model used to fit the measured responses was according to the Eq. 2:

where is the predicted response, is the model constant and are the linear coefficient, and are the quadratic coefficient, is the coefficient for the interaction effect, and and are independent variables. The goodness of the fit model was evaluated by the lack of fit test, the determination coefficient (), and the analysis of variance (ANOVA) using the Response Surface Regression (RSREG) procedure of SAS. Statistical fit of the model was determined by Fisher’s statistical test. The robustness of the model was assessed by the determination coefficient () and Fisher’s test at 95% confidence level.

Data processing and preparation activities: Data was collected in MS Excel files. Different tabs have been assigned for different measurements. For data presentation an index tab at the beginning of each Excel file was created with the sample nomenclature, an explanation of the content of the file and a description of each of the variables studied.