1. ABOUT THE DATASET

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Title: Data used in the article ‘Physical and chemical characterisation of conventional and nano/emulsions: influence of vegetable oils from different origin’

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Organisation: University of Reading

Rights-holder(s): Jansuda Kampa, Julia Rodriguez-Garcia (University of Reading)

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Description: This dataset contains data obtained from experimental work on the physical and chemical properties of oils, conventional and nanoemulsions. The data was obtained using a high-speed homogenizer, an ultrasound processor, a high-pressure homogenizer, a gas chromatographer equipped with a flame ionization detector (fatty acid composition), a spectrophotometer, a Chroma Meter, a rheometer (viscosity) and a dynamic light scattering (DLS) instrument (mean droplet diameter, polydispersity index and ζ-potential).

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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. ’PropertiesConventionalEmulsions’: this file contains data of the physicochemical analyses done in conventional emulsions in terms of:
	1. Table 1: Mean droplet diameter (MDD)
	2. Table 1: Polydispersity index (PDI)
	3. Table 1: ζ-potential
	4. Table 1: Colour
2. ‘StabilityConventionalEmulsions’: this file contains data of the analyses to assess the physical and chemical stability of conventional emulsions in terms of:
	1. Figure 2: Creaming index (CI)
	2. Figure 3: Thiobarbituric acid reactive substances (TBARS)
3. ‘PropertiesConvNanoEmulsions’: this file contains data of the physicochemical analyses done in conventional and nanoemulsions in terms of:
	1. Table 3: Mean droplet diameter (MDD)
	2. Table 3: Polydispersity index (PDI)
	3. Table 3: Thermal stability (TS)
	4. Table 3: Thiobarbituric acid reactive substances (TBARS)
4. ’PropertiesOils’: this file contains all the data of the physicochemical analyses done in the vegetable oils in terms of:
	1. Table S1: Fatty acid composition (% of total fatty acids)
	2. Table S2: Colour
	3. Table S3: Total phenolic content (TPC)
	4. Table S3: Free fatty acids (FFA)
	5. Table S3: Radical scavenging activity (DPPH)
	6. Table S3: Thiobarbituric acid reactive substances (TBARS)
	7. Table S3: Viscosity
	8. Table S3: Density

Variables explanation:

1. ’PropertiesConventionalEmulsions’:

• Extra virgin olive oil emulsion: EVO-E

• Cold pressed rapeseed oil emulsion: CPR-E

• Olive oil emulsion: OO-E

• Rapeseed oil emulsion: RO-E

• Sunflower oil emulsion: SO-E

• Lightness (L\*): 0(black) and 100 (white)

• Colour coordinate (a\*): -a\* (greenness) and +a\* (redness)

• Colour coordinate (b\*): -b\* (blueness) and +b\* (yellowness)

2. ‘StabilityConventionalEmulsions’:

• Extra virgin olive oil emulsion: EVO-E

• Cold pressed rapeseed oil emulsion: CPR-E

• Olive oil emulsion: OO-E

• Rapeseed oil emulsion: RO-E

• Sunflower oil emulsion: SO-E

1. ‘PropertiesConvNanoEmulsions’:

• Extra virgin olive oil emulsion: EVO-E

• Rapeseed oil emulsion: RO-E

• Sunflower oil emulsion: SO-E

4. ’PropertiesOils’:

• Extra virgin olive oil: EVOO

• Cold pressed rapeseed oil: CPRO

• Olive oil: OO

• Rapeseed oil: RO

• Sunflower oil: SO

• Total phenolic content values were expressed as gallic acid equivalents (mg GAE/ kg oil)

• Free fatty acids were expressed as percentage of oleic acid (C18:1)

• Lightness (L\*): 0(black) and 100 (white)

• Colour coordinate (a\*): -a\* (greenness) and +a\* (redness)

• Colour coordinate (b\*): -b\* (blueness) and +b\* (yellowness)

6. METHODS

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Data collection methods:

2.1.Reagents and Standards

Tween 20 (Polyoxyethylene sorbitan monolaurate), with hydrophilic-lipophilic balance (HLB) value of 16.7, was used as food-grade nonionic surfactant. Fatty acid methyl esters (the standard reference material FAMEs included C8:0, C10:0, C12:0, C14:0, C16:0, C18:0, C18:1, C18:2, C18:3), boron trifluoride reagent (BF3) (13–15% w/w in methanol), methanol (≥99.9% v/v) (CH3OH), heptane (99% v/v) (CH3(CH2)5CH3), sodium chloride (NaCl), Folin–Ciocalteu reagent, gallic acid (C6H2(OH)3COOH), sodium carbonate (Na2CO3), diethyl ether, ethanol (95% v/v) ((CH3CH2)2O), potassium hydroxide (KOH), phenolphthalein (C20H14O4), 1,1-diphenyl-2-picrylhydrazyl radical (DPPH), trichloroacetic acid (≥99% w/w) (CCl3COOH) and thiobarbituric acid (98% w/w) (C4H4N2O2S) were purchased from Sigma-Aldrich Co., Ltd. (Dorset, UK). Sodium hydroxide (NaHO), anhydrous sodium sulphate (≥99% w/w) (Na2SO4) and hydrochloric acid (37% w/w) (HCI) were purchased from Fisher scientific Co., Ltd. (Loughborough, UK). High-purity water was used for the preparation and dilution of reagents and standards.

2.2. Oil Samples

Five oils were selected for the study due to their high content of unsaturated fatty acids, their different unsaturation profile and extraction processes: 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), cold-pressed rapeseed oil (CPRO) (4.88% of saturated fat, 73.39% of monosaturated fat and 20.73% of polyunsaturated fat; Farrington’s mellow yellow brand, UK retail market), Olive oil (OO) (composed of refined olive oil and virgin olive oil, 13.28% of saturated fat, 79.73% of monosaturated fat and 6.98% of polyunsaturated fat; Brakes Bros Co., Ltd., , Kent, UK), rapeseed oil (RO) (refined rapeseed oil, 4.99% of saturated fat, 74.11% of monosaturated fat and 20.90% of polyunsaturated fat; Mazola brand, UK retail market) and sunflower oil (SO) (refined sunflower oil, 6.52% of saturated fat, 31.95% of monosaturated fat and 61.53% of polyunsaturated fat; Brakes Bros Co., Ltd., Kent, UK). The fatty acid composition of oils was analysed following the methodology described in Section 2.4.1. and is available in Table S1.

2.3. Emulsion Preparation

The conventional emulsion preparation procedure was based on the method described by Arancibia et al. [39] and Taha et al. [40] with some modifications. Conventional emulsions (oil droplet particle size of more than 200 nm) [35,36] were prepared in three steps. Firstly, a magnetic stirrer (Model SS3H, ChemLab, Zedelgem, Belgium) was used to disperse Tween 20 (5% w/w) in water (85% w/w) at 3.33 s−1 for 30 min at ambient temperature for complete dispersion. Then, the oil was added (10% w/w) to the aqueous phase during continuous stirring. Secondly, the emulsions were homogenised with a high-speed homogeniser (Model L4RT, Silverson, Bucks, UK) at 166.67 s−1 for 10 min. Thirdly, the emulsions were further processed in an ultrasound processor (32 mm diameter titanium probe, Model P100/6-20, Sonic Systems Limited, Ilmister, UK) at 100 watt, 20 kHz frequency, 5 microns amplitude at ambient temperature for 15 min (total power density delivered to the samples was 99 watts/L; power density was 12 watts/cm2). Then, the emulsions were left to cool down at ambient temperature and kept at 25 °C and 40 °C for 1, 7, 14, 21 and 28 days before measurements. All conventional emulsions were prepared in triplicate.

Nanoemulsions were produced after selecting three of the oils (EVOO, RO and SO). For nanoemulsion preparation, the coarse emulsions were processed in a high-pressure homogeniser (8.30H, Rannie, APV, Albertslund, Denmark) at 50,000 kPa for 1 cycle to produce droplet sizes < 200 µm. Then, the nanoemulsions were left to cool down at ambient temperature before measurements for 24 h. All nanoemulsions were prepared in triplicate.

The nomenclature used for the oil samples was as follows: extra virgin olive oil (EVOO), cold-pressed rapeseed oil (CPRO), olive oil (OO), rapeseed oil (RO), sunflower oil (SO). Conventional emulsion samples were named by adding a ‘-E’ after the oil name; nanoemulsion samples were named by addition of an ‘-NE’ after the oil name. When using the term ‘emulsions’, the authors refer to both conventional and nanoemulsions.

2.4. Physical and Chemical Properties of Oils

2.4.1. Fatty Acid Composition

The fatty acid composition of oils was determined according to Association of Analytical Communities method 969.33 [41]. Fatty acid methyl esters (FAMEs) were prepared by adding oils (200 mg) to a 0.5 M methanolic sodium hydroxide solution (4 mL). Then, the solutions were attached to a condenser and refluxed for 10 min until fat globules disappeared. Boron trifluoride solution (5 mL) was added, and the mixture continued boiling for 2 min. Heptane (5 mL) was added through the condenser, and the mixture was boiled a further minute. After boiling, the mixture was allowed to be tepid by keeping it at room temperature for 2 min, and saturated sodium chloride solution was added. Subsequently, the heptane layer was transferred into the test tube, and anhydrous sodium sulphate was added in order to remove the water. For the heptane phase, the solution was diluted with heptane to a 10% concentration, and 1µL was injected in a gas chromatographer (GC; Agilent 7890B gas chromatograph (Agilent Technologies Ltd., Didcot, UK) equipped with a flame ionisation detector (FID) and an HP-5 capillary column (30 m × 0.32 mm i.d., 0.25 µm film thickness) (Agilent Technologies Ltd., Didcot, UK) for analysis.

GC conditions were set up following the method described by Nhu-Trang et al. [42]. The column temperature was programmed from 70 °C (held for 3 min), then increased up to 166 °C at 3 °C/min rate and to 285 °C at 15 °C/min. Injector and detector temperatures were 250 °C and 300 °C, respectively. Split ratio injection was 1:50. Helium was used as carrier gas at a flow rate of 1.5 mL/min. The relative percentage compositions of fatty acids were computed by normalisation method from the GC peak areas and calculated as the mean value of three injections.

2.4.2. Total Phenolic Content

The total phenolic content (TPC) of oils was determined by using Folin–Ciocalteau colourimetric method according to Lee et al. [43] with some modifications. Oil samples (0.5 g) were extracted with methanol (10 mL) using a vortex mixer for 1 min. Then, 5 mL of the Folin reagent (previously diluted 10-fold with water) was added to 2 mL of the methanolic extract. The solution was incubated at room temperature for 5 min, followed by adding 7% (w/v) sodium carbonate (1 mL) and incubating for 90 min at room temperature. The absorbance was read at 725 nm using a spectrophotometer (CECIL, CE 1021, 1000 SERIES, Cambridge, UK). A calibration curve was constructed using gallic acid as standard, and the results were expressed as gallic acid equivalents (mg GAE/kg oil). A stock solution of gallic acid (0.5 mg/mL) was dissolved in 1 mL of methanol before diluting with distilled water to prepare a calibration curve of concentrations of gallic acid standards between 0.001 to 0.250 mg/mL. Measurements were taken in triplicate for each oil sample.

2.4.3. Free Fatty Acids

The free fatty acids were determined using the official cold volumetric titration method [44,45] with potassium hydroxide. The samples (2.5 g) were dissolved in 50 mL of neutralised mixture of diethyl ether and ethanol 95% (v/v). The mixture was titrated with potassium solution (0.1 mol/L) and using 0.3 mL of phenolphthalein solution (10g/L solution in 95% ethanol (v/v)) per 100 mL of mixture as an indicator. Measurements were taken in triplicate. Results were expressed as percentage of oleic acid (C18:1) and calculated using the following equation (Equation (1)):

FFA (%)=(V ×c ×M)/(10 ×m) (1)

where

V = the volume of titrate potassium hydroxide solution used, in millilitres,

c = the exact concentration in moles per litre of the titrated solution of potassium hydroxide used,

M = the molar weight in grams per mole of the acid used to express the result (oleic acid = 282) and

m = the weight in grams of the sample.

2.4.4. Emulsion Colour

The colour of the emulsions was assessed using a Chroma Meter CR-400 (Konica Minolta, Inc., Tokyo, Japan). The results were express in accordance with the CIELAB system with reference to illuminate D65 and a visual angle of 10°. The parameters determined were L\* (L\* = 0 (black) and L\* = 100 (white)), a\* (−a\* = greenness and +a\* = redness), b\* (−b\* = blueness and +b\* = yellowness). The samples were poured into a granular-materials attachment CR-A50 (Konica Minolta, Inc.,Tokyo, Japan) and measured in triplicate.

2.4.5. Determination of Radical Scavenging Activity

The antioxidant property of oils was evaluated by the 1, 1-diphenyl-2-picrylhydrazyl (DPPH) assay. The DPPH radical from the odd electron of nitrogen atom can be scavenged by receiving a hydrogen atom from antioxidant compounds [46]. When the DPPH stable free radical was reduced, a change of colour from violet to yellow was observed. The oil samples were analysed for their radical scavenging activity following the method described by Mraihi et al. [47] with some modifications. As a reagent, a 0.1 mmol solution of 1, 1- diophenyl-2-picry-hydrazyl (DPPH) was prepared in 80% methanol, and 5 mL of the solution was mixed with 0.25 mL of oil. This sample was incubated in the dark for 30 min. Then, the absorbance was measured at 517 nm against a control sample (5 mL of 0.1 mmol DPPH in methanol with 0.25 mL of blank solution). Measurements were taken in triplicate. The radical scavenging activity was expressed as the inhibition percentage of free radical DPPH, calculated using the following equation (Equation (2)):

DPPH·scavenging activity (%)=(1-As/Ac) ×100 (2)

where Ac is the absorbance of the control, and As is the absorbance of the sample.

2.4.6. Determination of Thiobarbituric Acid Reactive Substances (TBARS)

TBARS were determined according to the method of Qiu et al. [48] and Sharif et al. [49] with some modifications. Briefly, 0.1 mL of the oil sample 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 min, followed by centrifugation (Heraeus Multifuge 3SR Plus Centrifuge, Thermo Fisher Scientific Ltd., Waltham, MA, USA) at 10,000 g for 15 min. The absorbance of the supernatant was measured at 532 nm using a UV spectrophotometer (CECIL CE 1021 1000 Series, Cecil Instruments Ltd., UK). The concentrations of TBARS values were determined by using a standard curve prepared using 1,1,3,3-tetraethoxypropane (TEP) standard curve (coefficient correlation (R2) = 0.9994). TEP standards between 0.01 to 0.20 µg/ mL were prepared with trichloroacetic acid 7.5%. Three analytical repetitions of each measurement were performed for each emulsion batch.

2.4.7. Viscosity

The viscosity of oils was measured with a rheometer (MCR 102, Anton Paar, St Albans, UK) with a concentric cylinder (CC27, Anton Paar, St Albans, UK). The shear rate range used was from 0.1 s−1 to 200 s−1, and the temperature was maintained at 25 °C. Measurements were taken in triplicate. The viscosity values at 100 s−1 were taken for data analysis and comparison.

2.4.8. Density

The apparent density (weight by volume) of oil samples was determined by the method followed by Gunstone [5]. Oils’ weight and volume were measured at room temperature, and apparent density was calculated using the following equation (Equation (3)):

Apparent density (g/ml)= (Mass of oil (g))/(Volume of oil (ml)) (3)

2.5. Physical and Chemical Properties of Conventional and Nanoemulsions

2.5.1. 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) by following a method described in previous studies [49,50]. Emulsions were diluted 100-fold with deionised water and agitated in order to avoid multiple light scattering effects. The dispersion was decanted into polystyrene cuvettes, and the measurement was recorded at a wavelength of 633 nm at 25 °C. Measurements were taken in triplicate. The polydispersity index was calculated with the following equation (Equation (4)) [51]:

PDI =(〖σ⁄d)〗^(2 ) (4)

where σ is the standard deviation, and d is the mean particle diameter.

2.5.2. Creaming Index (CI) and Thermal Stability (TS)

Creaming index (%) was evaluated based on the method reported by previous studies with some modifications [39]. An amount of 10 mL of each emulsion was poured into a glass tube and stored at 25 °C and 40 °C in order to accelerate destabilisation mechanisms during storage. The total height (mm) of emulsion and cream layer were measured with a digital calliper after 1, 7, 14, 21 and 28 days. The Cl (%) was calculated using the following equation (Equation (5)):

Creaming index %=(Hc/Ht)×100 (5)

where Ht is the total height of the emulsion (mm), and Hc is the height of cream layer (mm).

Thermal stability was determined as described by Sahafi, Goli, Kadivar and Varshosaz [31]. Each emulsion (10 mL) was heated in a water bath at 80 °C for 30 min, followed by centrifugation at 1200× g for 10 min. The height (mm) of initial emulsion, cream layer and sedimentation phase was measured with a Digital Vernier Caliper. Emulsion thermal stability was calculated according to Equation (6):

Thermal stability %= ((HE-(HS+HC))/HE) ×100 (6)

where HE is the height of initial emulsion (mm), HS is the height of sedimentation phase (mm), and HC is the height of cream layer (mm).

2.5.3. Determination of Thiobarbituric Acid Reactive Substances (TBARS)

The measurement of TBARS was performed as explained in Section 2.4.6, with a different sample/reagent proportion [48]. An amount of 1 mL of emulsion was added to 5 mL thiobarbituric acid (TBA) solution and measured after 1, 7, 14, 21 and 28 days at stored temperature of 25 °C and 40 °C. Measurements were taken in triplicate.

2.6. Statistical Analysis

Statistical analysis of experimental data was performed using IBM SPSS 25 (IBM Corp, Armonk, NY, USA). One-way analysis of variance (ANOVA) and Tukey’s test at 95% confidence level (p < 0.05) were used to compare the mean values of viscosity, density, total phenolic components, free fatty acids, radical scavenging activity and TBARS of oil samples, and MDD, PDI, ζ-potential, creaming index and TBARs of emulsion samples. Moreover, to evaluate the effect of storage time in conventional emulsions, a two-way analysis of variance and Tukey’s test at 95% confidence level (p < 0.05) were conducted. The interaction of the two independent factors, oil type (EVOO, CPRO, OO, RO, SO) and storage time (1, 7, 14, 21 and 28 days), at two different storage temperatures (25 °C and 40 °C), was evaluated for the creaming index and TBARS values of conventional emulsion samples. Pearson correlation was calculated for the physical and chemical properties of vegetable oils, conventional, and nanoemulsions at 95% and 99% confidence level (p < 0.05 and 0.01, respectively). The correlation coefficient (r) was obtained: very weak correlation (0.01 ≤ r < ± 0.10), weak (± 0.10 ≤ r < ± 0.50), moderate (± 0.50 ≤ r < ± 0.80), strong (± 0.80 ≤ r < ≤ 1.00) and perfect (r = ± 1.00) [52].

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.