



Optimization of a gelled emulsion intended to supply ω -3 fatty acids into meat products by means of response surface methodology



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ABSTRACT

The optimization of a gelled oil-in-water emulsion was performed for use as fat replacer in the formulation of ω -3 PUFA-enriched cooked meat products. The linseed oil content, carrageenan concentration and surfactant-oil ratio were properly combined in a surface response design for maximizing the hardness and minimizing the syneresis of the PUFA delivery system. The optimal formulation resulted in a gelled emulsion containing 40% of oil and 1.5% of carrageenan, keeping a surfactant-oil ratio of 0.003. The gel was applied as a partial fat replacer in a Bologna-type sausage and compared to the use of an O/W emulsion also enriched in ω -3. Both experimental sausages contributed with higher ω -3 PUFA content than the control. No sensory differences were found among formulations. The selected optimized gelled oil-in-water emulsion was demonstrated to be a suitable lipophilic delivery system for ω -3 PUFA compounds and applicable in food formulations as fat replacer.

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1. Introduction

The characteristics of fat analogues intended to replace animal fats are needed in order to achieve the appearance and the technological, rheological and sensory properties required for use in the food industry (Tye, 1991). In fact, the use of fat replacers can cause, in some cases, technological problems due to the fact that fat has a great impact on flavor, palatability and texture of foods (Delgado-Pando, Cofrades, Ruiz-Capillas, & Jimenez-Colmenero, 2010; Horita, Morgano, Celeghini, & Pollonio, 2011; Hort & Cook, 2007).

The use of emulsion based delivery systems is a suitable technology for protection and release of lipids in food (McClements, Decker, & Weiss, 2007; Salminen, Herrmann, & Weiss, 2013). There has been an increasing interest in improving the functional performance of foods using a wide variety of novel types of emulsion delivery systems, including solid lipid particles, filled hydrogel particles and conventional, multiple and multilayer emulsions (McClements, 2010). These systems are able to incorporate lipophilic functional agents with beneficial health effects into food products (Chung, Degner, & McClements, 2013; Nielsen & Jacobsen, 2013; Poyato et al., 2013; Taneja & Singh, 2012; Valencia, O'Grady, Ansorena, Astiasarán, & Kerry, 2008). Some of these emulsion delivery systems have been used as fat replacers to produce high ω -3 products for improving the nutritional quality of new products. In this sense, the potential development of functional meat products using reformulation strategies has been attempted with the aid of emulsion based systems. The substitution of pork back

fat with pre-emulsified oils ω -3 type PUFA oils has been demonstrated to be a good strategy to achieve healthier lipid profiles in these products (Berasategi et al., 2011; Garcia-Iniguez de Ciriano et al., 2010).

Recently, some papers (Jiménez-Colmenero, Triki, Herrero, Rodríguez-Salas, & Ruiz-Capillas, 2013; Salcedo Sandoval, Cofrades, Ruiz-Capillas Pérez, Solas, & Jiménez-Colmenero, 2013; Triki, Herrero, Jiménez-Colmenero, & Ruiz-Capillas, 2013a,b; Triki, Herrero, Rodríguez-Salas, Jiménez-Colmenero, & Ruiz-Capillas, 2013) have used konjac gel and oil stabilized in a complex konjac matrix as potential fat analogues to reduce or improve the lipid fraction of different meat products, obtaining good results.

In comparison to oil-in-water emulsions, gelled emulsions could be a better option to mimic hardness and water holding capacity of pork back fat used in most of the currently consumed meat products.

The objective of our research was to optimize the formulation of a gelled oil-in-water emulsion prepared with oil rich in ω -3 fatty acids (linseed oil), carrageenan, a surfactant and water, in order to obtain a successful functional ingredient by means of a factorial design of response surface. This optimized gelled oil-in-water emulsion was used as partial fat replacer in a meat product (Bologna type sausages), and its nutritional, sensory and technological properties were assessed.

2. Material and methods

2.1. Materials

Fresh pork meat (shoulder and front leg) and back fat were obtained from a local meat market. The meat was trimmed of visible fat and connective tissue. Linseed oil (Biolasi Productos Naturales, Guipúzcoa,

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Spain) was obtained in a local market. BDRom Carne (a mixture of typical aromatic compounds) and the red colorant Carmin de Cochenille 50% (E-120) were obtained from BDF Natural Ingredients S.L. (Girona, Spain). Carrageenan (kappa-carrageenan) was kindly donated by Cargill (San Sebastián, Spain) and Curavi (a mixture of curing agents: NaCl, E-250, E-252 and antioxidant E-331) BHA, polyphosphates, monosodium glutamate, sodium ascorbate and garlic were kindly donated by ANVISA (Arganda del Rey, Madrid, Spain). All the chemical reagents and Polysorbate 80 were obtained from Sigma-Aldrich Chemical Co. (MO, USA).

2.2. Gelled emulsion design

Response surface methodology (RSM) was applied to optimize the formulation of an oil-in-water gelled emulsion. The effect of three independent variables including oil concentration, carrageenan concentration and surfactant–oil ratio (SOR) were studied in order to maximize hardness and minimize syneresis of the obtained gels. The first approach for the optimization was the delimitation of the ranges for the three ingredients used in the preparation of the gels. The maximum oil concentration technologically able to produce a gelled oil-in-water emulsion was selected as the upper limit for this ingredient (70%), whereas the lowest limit (40%) was the minimum oil content needed for achieving a significant amount of fatty acids based on nutritional value. The lowest (0.5%) and upper (1.5%) limit for carrageenan concentration were the minimum and maximum carrageenan concentration able to form a gelled oil-in-water emulsion with the lowest and highest amount of oil, respectively. In the case of polysorbate 80, the limits were expressed as the ratio between surfactant and oil amount (SOR). The lowest limit for SOR was that needed for obtaining a stable gelled oil-in-water emulsion (0.003), whereas the upper limit was the maximum concentration whose bitterness was not detected in the gelled emulsion formed (0.005).

Taking into account these limits, the application of the central composition design ($2^3 + \text{star}$, including 2 central points, Statgraphics Centurion XV software), resulted in a design of 16 experimental settings, which were carried out in triplicate, and in random order (Table S1, supplementary material).

2.3. Gelled emulsion preparation and analysis

50 ml of every 16 types of gelled emulsions were prepared as follows: the oil phase containing the hydrophobic surfactant (Polysorbate 80) was added to the aqueous phase that included the corresponding percentage of carrageenan and homogenized. Both phases were previously heated separately to 70 °C. After the homogenization process (16,000 rpm, Ultra-Turrax® T25basic), the emulsions were cooled to room temperature in a sealed flask, allowing the k-carrageenan to polymerize. The gels were kept overnight under refrigeration (4 °C) before analysis.

For the determination of hardness and syneresis, gel samples were cut into cylinders ($D = 2.8 \text{ cm}$, $h = 1 \text{ cm}$). Hardness was measured using a universal texture analyzer (TA-XT2i, Stable Micro Systems, Surrey, United Kingdom) with a P 0.5R probe to determine the textural characteristics of gels. Cylindrical samples were placed under the probe and underwent compression under a 5 kg load cell at a deformation rate of 30%. Force–time curves were recorded at a crosshead speed of 0.5 mm/s. Ten measurements were performed in each type of sample.

For the determination of syneresis, each sample was weighed (W_0) inside Petri dishes, and placed in a cabinet at 25 °C for 3 days. The water that condensed on the container walls was removed before weighing the gels (W_t). The syneresis of the gels was calculated as follows: syneresis (%) = $[(W_0 - W_t) / C_0] \times 100$, where C_0 is the initial water content in the sample, expressed in percentage. The experiment was performed in triplicate.

The application of the multiple response optimization to hardness and syneresis results let us to conclude that the optimum combination of the gel ingredients was: 40% oil, 1.5% carrageenan and 0.003 SOR. This was the gel used as partial fat replacer in Bologna type sausages elaborated in the second part of the work.

2.4. Sausage formulation and processing

Three different formulations (Table S2, supplementary material) of Bologna-type sausages were manufactured in a pilot plant according to the procedure described by Berasategi et al. (2011). Control products (control) contained 16% pork back fat, whereas in the two experimental batches, half of the pork back-fat was substituted by a conventional oil-in-water emulsion (emulsion) or by the previously optimized gelled oil-in-water emulsion (gel) rich in ω -3 fatty acids. The conventional oil-in-water (O/W) emulsion was prepared according to the procedure described by Garcia-Iniguez de Ciriano et al. (2010), and the gelled oil-in-water emulsion was prepared as previously described (Section 2.3). The conventional and gelled emulsions were kept under refrigeration until their use.

Previous experiments (Berasategi et al., 2011) demonstrated the need for the addition of extra antioxidants when cooked meat products contained high PUFA fat sources. Thus, in both experimental batches, 200 mg of BHA/Kg meat batter were added in the mixture of all additives. The control type was manufactured free of extra antioxidants. The formulations were carried out in triplicate. Additionally, samples from every type of formulation were stored under refrigeration (4 °C) for 35 days.

2.5. Analysis of sausages

Color of sausages was measured using a digital colorimeter (Chromameter-2 CR-200, Minolta, Osaka, Japan) to obtain the color coordinates L^* , a^* and b^* . These values are used to calculate the Euclidean distance value ($\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$) of products along the storage. The texture (TPA) was measured using a Universal TA-XT2i texture analyzer. Conditions applied for color and texture were those described by Berasategi et al. (2014a).

The method of Folch, Lees, and Stanley (1957) was used for the extraction of fat.

In order to assess the oxidation status of the Bologna-type sausages, TBARS (thiobarbituric acid value) value was determined in all three types of sausages over storage time, using 0.25 g fat, according to the method described by Maqsood and Benjakul (2010) with slight modifications (Poyato, Ansorena, Navarro-Blasco, & Astiasarán, 2014). Results were expressed in mg of malondialdehyde (MDA) equivalents/kg sausage.

The fatty acids were determined in the lipid extracts by gas chromatography FID detection according to the procedure described by Valencia et al. (2008). Moisture, protein and fat content were analyzed using official methods (AOAC, 2002a,b,c).

Fat extraction, TBARS, color and texture were measured every 7 days of storage.

2.6. Sensory analysis of meat products

A triangle test was performed to determine the existence of perceptible sensory differences in hardness, taste and appearance between control and the gel containing products (gel) and between the two experimental products (emulsion and gel). A total of 21 semi-trained panelists participated in the sessions. Three samples, of which two were identical, were presented to each panelist, and they were asked to indicate which sample differed from the others. The number of correct answers was collected. According to the Spanish norm UNE

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