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# Microencapsulation of extracted bioactive compounds from brewer's spent grain to enrich fish-burgers

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ABSTRACT

In this work bioactive compounds (polyphenols and flavonoids) were extracted by supercritical $CO_2$ from brewers's spent grain (BSG). The extract was microencapsulated to mask its unpleasant and bitter taste and tested on fish-burger formulation. The extraction process was carried out at 40 °C and 35 MPa of pressure whereas the microencapsulation was performed by means of a spray-drying, using Capsul® as wall material and modifying inlet temperature (90–120–150 °C) and ratio between extract and carrier (1:2; 1:4; 1:6; 1:8). A sensory evaluation of fish-burgers prepared with different bioactive powders of BSG was carried out to establish the best combination of operating parameters. The sample with 5% microencapsulated BSG extract and Capsul® solution in ratio equal to 1:2 at 150 °C was chosen as the best compromise between chemical properties of active powder and sensory evaluation of fish sample. Finally, the antioxidant properties of fish burger with microencapsulated BSG extract were compared to the control. Results confirmed the potential use of BSG as food ingredient because fish-burgers were found richer in polyphenols (30%) and flavonoids (50%) and with a better antioxidant activity than the control sample.


## 1. Introduction

During the wort elaboration step in the production of beer, several by-products are generated. Brewer's spent grains (BSG) are the most common and abundant ones. This material is usually used as animal feed, composted or disposed as landfill (Mussatto et al., 2006; Jay et al., 2008). However, alternative and valorized uses are sought after, both due to the increased cost of its disposal and considering also its high content in bioactive components, such as phenolic acids, flavonoids, vitamins and minerals, widely recognized to have important antioxidant and antiradical properties (Goñi and Hervert-Hernañdez, 2011). Additionally, scientists, producers and consumers consider BSG of great interest for food industry (Naczk and Shahidi, 2004). In fact, Prentice et al. (1978) and Stojceska et al. (2008) have shown that BSG can be effectively integrated into ready-to-eat snacks and into cookie formulation to increase dietary fiber, crude protein and fat content. Nowadays there is a growing interest in the recovery of vegetable by-products and their conversion into high-value products to obtain functional compounds because of a constant research to find new alternatives regarding the food fortification.

However, the application of BSG to food is still limited, since it can impart unpleasant flavors and aromas (Townsley, 1979). Bakowska et al. (2003) have studied the stability of polyphenols, proving that they are influenced by pH, metal ions, light exposure, temperature, oxygen and enzymatic activities. Therefore, to fully exploit the potential of BSG in food products, is necessary to identify a way to mask the undesirable attributes and simultaneously preserve the stability of polyphenols or other bioactive compounds.

Spray drying could be an appropriate and valid technique for minimizing these problems; furthermore, it is very easy to industialize and allows for continue production (Su et al., 2008). Generally, with microencapsulation technologies the easily degradable ingredients are incorporated within a coating or wall material that protects the components against environmental stresses (Bakowska-Barczak and Kolodziejczyk, 2011). Among the numerous wall materials or encapsulating agents that are available for food application, a chemically

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modified starch (Capsul) was used for this study. Capsul shows low viscosity, good film-forming properties and thermo-protective effect during the exposure to high temperatures (Reineccius, 1991; Shahidi and Han, 1993; Marchal et al., 1999). According to a previous work, it appeared to be the better material to microencapsulate propolis extract during atomization, if compared to arabic gum (Spinelli et al., 2015).

The acquisition of bioactive compounds requires their extraction from by-products of beer without altering their natural original properties. Several techniques for extracting the antioxidant components from BSG have been developed (Mussatto, 2009). In general, the use of basis of either acid hydrolysis or saponification (with 1–4 M NaOH) and liquid–liquid or liquid–solid extraction with polar solvents are the most common methods used to recover natural antioxidants from BSG (McCarthy et al., 2013). Alternatively, supercritical fluid extraction is becoming a promising option. In fact, in our previous work we have been identified the best operating conditions to extract the maximum concentration of total phenols, flavonoids and free antiradical scavenging activity from BSG. This used supercritical fluid extraction (SFE) with carbon dioxide ($CO_2$), as supercritical solvent, and ethanol (EtOH), as co-solvent. The following process variables were used to optimize the active compound extraction: temperatures (40–50–60 °C), pressure (15, 25 and 35 MPa). Moreover, several ethanol concentrations were also used (20, 40 and 60% ethanol (v/v)) (Spinelli et al., 2016). Based on the best SFE conditions (40 °C, 35 MPa and 60% ethanol) previously identified to achieve a high phenolic and flavonoid content and good antioxidant properties from BSG, the objective of the present study was to identify the best spray drying conditions to obtain a powder able to mask the unattractive attributes of extracts. In particular, the microencapsulation by spray drying of polyphenols and flavonoids extracted from BSG was conducted in order to obtain a positive sensory evaluation in a proper fish-burger formulation. Finally, the antioxidant properties in the optimized fish-burgers with microencapsulated BSG extract were also addressed.

## 2. Materials and methods

### 2.1. *Raw materials and chemicals*

Brewer's spent grains (BSG) were supplied by a local brewery industry located in Puglia (EBERS, Foggia, Italy). They were dried overnight in a dryer (T2—Namad, Rome, Italy) at 35 °C reaching a moisture accounting for 5.62 ± 0.18% (according to AACC (2000) method 44-19). The median particles size (by mass) was determined by sieving method and it was found to be about 500 μm.

As carrier of spray drying, Capsul (C), a chemically modified starch (Ingredion Incorporated, Westchester, USA) was used. Folin–Ciocalteu reagent, anhydrous sodium carbonate ($Na_2CO_3$), gallic acid monohydrate, sodium nitrite ($NaNO_2$), aluminum chloride ($AlCl_3$), sodium hydroxide (NaOH), quercetin, DPPH radical were supplied by Sigma-Aldrich (Milan, Italy). Food grade ethanol (EtOH) was provided by Perrin's Chemicals (Triggiano, Italy). Sapio (Monza, Italy) supplied $CO_2$ with purity degree of 4.5 for SFE.

### 2.2. *Supercritical fluid extraction*

The extraction process was carried out using the supercritical fluid extractor *Speed* SFE-2 (Applied Separation, Allentown, USA), capable of pressures up to 69.0 MPa and temperatures up to 240 °C, static and dynamic extraction with flow from 0 to 10 L/min (gaseous $CO_2$ at atmospheric pressure) and extraction vessels from 1 to 20 L. The unit includes two pumps: a solvent pump that delivers the fluid throughout the system, driven by air compressed obtained from a compressor, and a modifier pump for the addition of organic co-solvent. The system was set to the desired temperature (40 °C) and pressure (35 MPa) with suitable switches, while a metering valve was used to vary the $CO_2$ flow rate. The $CO_2$ flow rate used was set at 2 L/min, whereas the collection condition was at room temperature and atmospheric pressure. The process lasted 240 min. Briefly, 35 g of BSG were loaded into the 50 mL extraction vessel with diameter of 15 mm that was placed in the extractor and was allowed to equilibrate to the desired temperature. Upon reaching the desired temperature, pressurization was initiated and the fluid ($CO_2$ + 60% ethanol, v/v) flowed through the extraction vessel from the bottom to the top. The extract-laden gas from the extractor was passed through a heated metering valve, where the supercritical $CO_2$ was depressurized, and the extract was collected in a separator vessel while $CO_2$ was vented by a flow meter. The extract was placed overnight in vacuum oven at 30 °C in order to remove ethanol. The residue was dissolved in 20 mL of absolute ethanol and filtered through a 0.45 μm syringe filter (Teknokroma PTFE 0.45 μm, Sant Cugat del Vallés, Barcelona, Spain). To obtain an appropriate quantity of extract (E_BSG), several extractions were carried out.

### 2.3. *Microencapsulation of BSG*

A mini Spray Dryer B-290 (BÜCHI Labortechnik AG, Flawil, Switzerland) was used to microencapsulate BSG extract. At first, Capsul (C) was dissolved in distillated water at the concentration of 30 g/100 mL, named carrier solution. The BSG extract was added to the carrier solution at proportions of 1:2, 1:4, 1:6 and 1:8 (w/w) and homogenized by an Ultra Turrax IKA T25 basic homogenizer (IKA Works Inc., Staufen, Germany) for 2 min at 15,000 rpm. The resulting formulations were spray dried at different inlet drying air temperatures (90, 120, 150 °C). The aspiration rate and pump flow used for all the experiments were fixed at 100% and 25%, respectively. At the end of each drying session, the powders were collected, placed in closed vials and kept at room temperature in a dry and dark place until analysis.

Samples were labeled using the format SDX/Y (e.g. "SD2/90") where X refers to the ratio of carrier solution to BSG extract and Y refers to the inlet spray drying temperature in °C.

The microencapsulation process was conducted in triplicate.

### 2.4. *Fish-burger preparation*

Fresh sea bass fillets (*Dicentrarchus labrax*) were obtained from a local seafood company (Tortuga s.r.l., Manfredonia, Italy). On arrival at the laboratory, they were trimmed to remove bones and skin and stored at −20 °C until use. In accordance with the recipe optimized in a previous work (Spinelli et al., 2015), fish-burgers of 40 g were prepared using minced fish mixed with 5% of spray-drying powders, 10% potato flakes (Digei s.r.l., Foggia, Italy) and 9% extra virgin olive oil (Olearia Desantis s.p.a, Bitonto, Bari, Italy). The fish-burgers were produced in moulds with a diameter of 5 cm and a height of 1 cm. Thus, fish-burgers were cooked in an electric convention oven (H2810, Hugin, Milan, Italy) at 180 °C for 15 min. Three independent replicates were used for each fish-burger formulation. Fish-burgers were labeled using the format FBX/Y (e.g. "FB2/90") where X refers ratio of carrier solution to BSG extract and Y refers to the inlet spray drying temperature to produce different powders. In addition, a fish-burger control (CTRL) was composed by minced sea bass enriched with 5% of wall mate-