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## Reorganisation of starch, proteins and lipids in extrusion of oats

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#### A R T I C L E I N F O

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#### ABSTRACT

The effects of extrusion temperature, screw speed and specific mechanical energy (SME) on reorganisation of oat components, starch, proteins and lipids, during extrusion of whole oat flour were studied. High SME transformed whole oat flour to a more homogenous matrix. However, based on CLSM images, high SME induced lipid separation from the oat matrix in high screw speeds but not at low extrusion temperatures. Cellular structures were broken and degraded and water solubility of oat components increased. Minimum extrusion temperature of 110 °C and screw speed of 200 rpm was needed for complete melting and solubilizing oat starch whereas cell wall polysaccharides were solubilized already at low extrusion temperatures due to high friction. Oat proteins, globulins, were only partially denaturated even in extreme conditions (130 °C), but their solubility in water decreased substantially in milder conditions. Furthermore, extrusion inactivated endogenous lipases effectively already in mild extrusion conditions (770 °C). Native amylose-lipid complexes were partially destroyed in extrusion.

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### 1. Introduction

The food extrusion process includes application of heat, moisture, shear and pressure to structures built of biopolymers, such as starch, protein and lipids, usually originating from cereals, leading to formation of a viscoelastic melt in the extruder barrel (Colonna et al., 1989). During extrusion, biopolymers undergo many chemical and physical transformations Such as loss of crystallinity and depolymerisation of starch, denaturation and cross-linking of proteins, complexation between amylose and polar lipids and degradation reactions of polymers and other molecules. Furthermore, the enzymes are inactivated, which inhibits food deterioration during storage. Extrusion may also destroy naturally occurring toxic substances and increase microbial stability (Colonna et al., 1989; Mitchell and Areas, 1992).

Variations in extrusion process parameters such as temperature, feed moisture, feed rate, screw speed and the addition of lipids have major impacts on the physicochemical properties of extrudates (Singh et al., 1998). The mechanical forces, which are varied by the operating conditions for the extruder, experienced by the material, are often considered the critical factor in the alteration of the biopolymers. This mechanical energy is usually quantified by a measure of the specific mechanical energy (SME). While cereal extrusion with wheat and maize has been studied extensively (Abu-Hardan et al., 2011; Carvalho and Mitchell, 2000), much less attention has been paid to oat extrusion (Gutkoski and El-Dash, 1999; Yao et al., 2011). Oats are an interesting cereal as they contain significant

bats are an interesting terear as they contain significant amounts of the dietary fiber β-glucan, protein with high nutritional value compared to other cereals and unsaturated lipids (Lásztity, 1996). Processing of oats are challenging due to the high amounts of these lipids and fiber, which affect both rheology and stability. Lipolytic enzymes in oats are 10–15 times more active than those of wheat (Matlashewski et al., 1982). Thus, inactivation of lipid hydrolysing enzymes (mainly lipases) must be carried out to avoid rancidity and bitter taste. Most of the lipids in oat grain are located in the bran and the starchy endosperm. Endosperm contains about 80–90% of the lipids in oat groat, which makes it unique compared with other cereals, where only around 50% of lipids are located in this fraction (Wicklund and Magnus, 1997).

In addition to inactivation of lipases, reorganisation of oat macromolecules within the high shear regimes occurring during





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Abbreviations: CLSM, confocal laser scanning microscopy; DSC, differential scanning calorimetry; RVA, rapid viscosity analyser; SME, specific mechanical energy; WAI, water absorption index; WSI, water solubility index.

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extrusion may play a role in lipid stability. In flour, the biological superstructures, which contain the storage macromolecules of oat grains, may have been partially lost during milling. Within thermomechanical extrusion, even greater destruction of the structures of all the components could be expected to occur. Starches often play a key role in cereal extrusion where they form the continuous melt phase and therefore their conversion has been extensively studied. Changes to the starch that are expected to occur, depending on process conditions, include: expansion and breakage of starch granules, crystallinity changes, release of amylose and amylopectin from the granule structure into a homogenous melt (Lai and Kokini, 1991). Extrusion is performed usually in limited water conditions (5-40%). High molecular weight polymers remain mostly intact, but depending on the conditions, the highly branched amylopectin could be expected to reduce in size (Lillford, 2008). Batterman-Azcona et al. (1999) studied the effect of SME on protein bodies and  $\alpha$ -zeins in corn flour extrudates and found that under mild processing conditions, protein bodies remained intact, but under harsher conditions, protein bodies were disrupted and  $\alpha$ -zein was released. These  $\alpha$ zeins were aggregated due to hydrophobic interactions and disulphide bonds. As amphiphilic components, proteins could effectively emulsify lipids and thus increase their oxidative stability during storage.

Reorganisation of the biopolymer matrix upon processing and its consequences to localisation of lipids need to be understood in order to control oxidation in extruded oats and in particular, their naturally occurring lipids. The aim of the present work was to study changes in whole oat flour due to extrusion: disintegration and rearrangements of biological superstructures as well as molecular level changes in starch, proteins and lipids. Effects of extrusion temperature and screw speed were studied in relation to microstructure, thermal transitions, viscosity and solubility of the extrudates.

#### 2. Materials and methods

#### 2.1. Materials

Native oat grains from Raisio Group (Nokia, Finland) were used as raw material for extrusion experiments. Oat protein concentrate was made from whole oat flour by defatting it using supercritical CO<sub>2</sub> extraction. After lipid extraction, the powder was air-classified and concentrated oat protein fraction was collected, resulting in a total 59% protein.

#### 2.2. Compositional analyses

#### 2.2.1. Lipid determination

Total lipids were extracted after HCl hydrolysis of the milled samples with petroleum ether and diethyl ether according to the AOAC official method 996.06 (AOAC, 2001). Fatty acid contents of the extracts were analysed after methylation of the samples by GC as presented earlier by Damerau et al. (2014). Total lipid contents were calculated as the sum of fatty acids 16:0, 18:0, 18:1, 18:2 and 18:3 (mean  $\pm$  standard deviation).

#### 2.2.2. Protein content determination

Protein content of the milled samples were analysed using the Kjeldahl method. The nitrogen content was converted to protein by multiplying with a factor of 6.25. Soluble protein was determined from the sample solution using the Bio-Rad DC protein assay kit (Bio-Rad, Richmond, USA) based on reaction of protein with an alkaline copper tartrate solution and Folin reagent. The characteristic blue colour of the reaction was measured spectrophotometrically at 750 nm absorbance wavelength. Protein concentration was calculated using bovine albumin serum as a standard.

#### 2.2.3. Total and soluble starch determination

Total starch (including resistant starch, maltodextrins and Dglucose) from the milled samples was determined using Megazyme (Megazyme, Wicklow, Ireland) total starch assay kit which is based on hydrolysing starch to glucose with thermostable  $\alpha$ -amylase and amyloglucosidase enzymes. Amount of total starch was then quantitatively measured in a colorimetric reaction of glucose using a spectrophotometer at 510 nm absorbance wavelength. Soluble starch was determined in the same way as total starch, excluding  $\alpha$ amylase treatment.

#### 2.3. Pre-treatments and extrusion of oats

Impact hulling was used to remove the hulls. Dehulled whole grain oats were milled with a Fritsch cutting mill using 4 mm screen and ground material was extruded within 4 h after milling.

Whole oat flour was extruded with a Thermo Prism TSE24MC twin screw extruder (Thermo Fisher Scientific Ltd). The extruder was equipped with a gravimetric feeder and a water pump, which were used to control the solid feed and the water input. For all samples, the water feed rate was 0.48 kg/h and a flour feed of 4 kg/h was used, resulting in an initial water content of 19.4%. Screw configuration contained two mixing elements at the die end and all the rest of the elements were feed screws. The extruder barrel consisted of 10 adjustable heating blocks. The temperature within the barrel was 80 °C for three blocks at the feed end and the temperature of the seven blocks at the die end was equivalent with the die. The extruder was fitted with a 30 \* 1 mm slit die. During the extrusion process, the barrel temperatures, screw speed, screw torque and feed rate were monitored and when they were stabilised the representative samples were collected. Extruded ribbons were oven-dried at 70 °C for 15 h.

Two sample series were created having seven samples in each series. In the first series the die temperature was changed from 70 to 130 °C, while screw speed was kept constant at 200 rpm. In the second series, screw speed was altered between 100 and 400 rpm while die temperature was kept constant at 110 °C.

Specific mechanical energy (SME), which is generally used as the main parameter to evaluate extrusion performance, was calculated with the following equation according to Abu-Hardan et al. (2011):

Calculated SME values are presented in Table 3.

(1)

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