Feed compositional effects on rheological and microstructural characteristics of high protein vegetable extrudates

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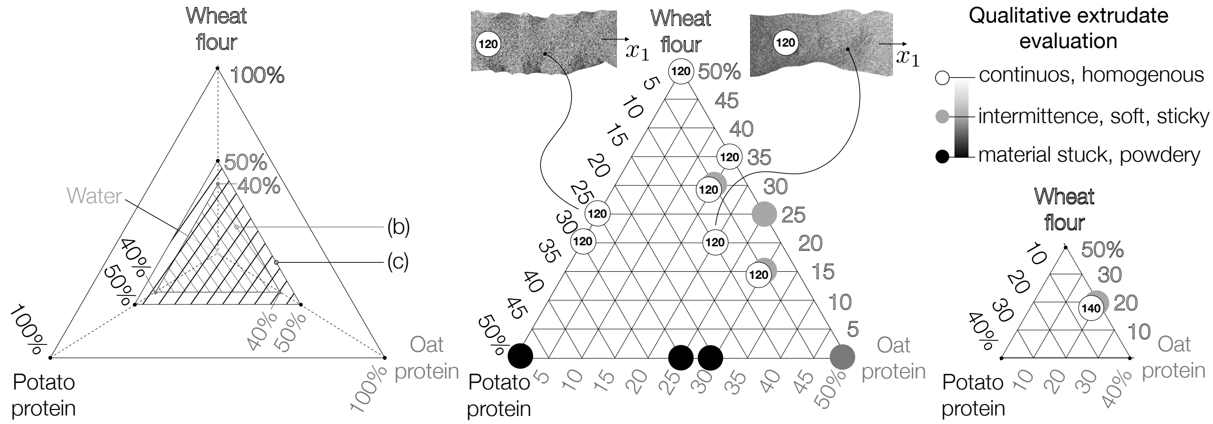
ABSTRACT

In this study, novel high protein biopolymeric melts were quantified by extrusion inline rheological measurements and the microstructure of the extrudates was characterized with optical microscopy. The raw ingredients, i.e. feed composition, used consisted of ternary protein mixtures, i.e. oat, what and potato proteins, together with potato starch and potato fibers mixed in different combinations. The rheological response of the high protein vegetable mixtures showed shear thinning behavior independent of the feed composition and moisture content in the range of shear rates investigated. The experimental data fitted well with power law model and the variation of the model parameters was interpreted based on the feed composition and resulting microstructure of the extrudates. Microstructural investigations revealed protein and fibre agglomerates while starch formed a continuous phase.

INTRODUCTION

The call for novel food materials capable of substituting meat products is strongly substantiated by the increased world human population growth, environmental issues as well as personal health considerations.[1,2] One of the greatest threats to our environment has been highlighted as being the meat industry, contributing to 14% of greenhouse gas emissions.[1] The importance for the future of sustainable living is however met with significant challenges to be overcome in developing and processing novel food formulations. Waste from the agro-industry commonly, used for animal feed, is currently considered as a potential sustainable source for human food. Some protein sources from the waste can be used for natural and highly nutritious food products. However, even if from nutritional point of view such food systems are desired, their behavior during processing significantly hinders the development of novel food formations. In this respect, extrusion / extrusion cooking stands out as an emerging technique within the food industry, offering unique possibilities for the development of new food formulations in terms of mixing, heating, texturing and shaping.[3,4] Such novel systems, however, can exhibit complex behavior when subjected to flow and deformation due to their complex structure and subsequent physicochemical changes occurring during food extrusion.[3,5,6] Thus, food extrusion of novel waste-based products is most often accompanied by surface dryness, rough surface, the extrudate is too wet, sticky and/or soft, or brittle and hard, the material gets stuck inside the flow system stuck, or a powdery extrudate is obtained, the extrudate exhibits instabilities etc.. Preliminary trial tests on the extrusion of novel high protein content ternary blend formulations based waste products, highlighting the qualitative extrudate evaluation, is presented in Figure 1. The protein blend composition is represented as a quaternary diagram corresponding to wheat, oat, potato as proteins and water as plasticizer, Figure 1(a). The processing conditions were determined based on trials aimed at establishing their potential as novel food products. The complexity of the system as well as the lack of understand their flow and



(a) (b) (c)

Figure 1. Feed composition of quaternary compositions containing ternary (waste) protein mixtures of what oat and potato and water as plasticizers, (a), and qualitative evaluations of their processing through extrusion for two moisture contents (b) 50wt% and (c) 40wt%. In the cases of continuous homogenous extrudates obtained, the extrusion temperature is specified.

deformation, i.e./ rheological properties, significantly hinders the design of their extrusion cooking. When considering in-situ qualitative extrusion flow observations, radically different flow behaviors are observed depending on the particular protein source tested. In Figure 2 two flow scenarios are presented during the extrusion flow via a compression screw (compression ratio 2:1). The in-situ observations were performed by stopping the extrusion process and immediately extracting the screw out of the barrel. Thus, Figure 2(b) reveals a dispersed plug flow[7] whereas Figure 2(c) a bifurcating unwrapped screw flow showing the onset of secondary flows with the growth of the melt pool. The two scenarios encountered highlight the importance of understanding the thermo-rheological behavior of food systems in order to tailor their feed composition and processing conditions to desired product properties. Furthermore, adding to the complexity of the problem, proteins of different sources are a requirement from nutritional point of view. Preliminary trails on ternary protein compositions containing what, oat and potato proteins, i.e. Figure 1, have revealed however limitations in their potential use as food products due to their poor water survivability. Thus, for optimal product properties, the compositions have to be augmented by additional components.

From rheological point of view, in the most simple case, such food compositions are characterized by a (shear) viscosity function of the form $\eta = \eta(T, \dot{\gamma}, t)$, where T is the temperature, $\dot{\gamma}$ is the shear rate and t is the time. The most suitable rheological characterization method must thus be based on inline (extrusion) rheometry. Inline rheological measurements during extrusion offers the possibility of determining the viscosity function at a constant deformation and thermal history, i.e. processing history, and at processing shear rates. In contrast, capillary rheometry is characterized by a variable residence time distribution[8] whereas in rotational rheometry a moisture control system must be available and the measurements would be limited to low shear rates. Inline rheological measurements are thus critical to understand the flow of such food systems. In this framework we present in this publication preliminary inline viscometry during the extrusion of novel high protein content mixtures highlighting the influence of feed compositional effects.

To summarize, two fundamental issues in the development of novel high protein mixtures based on food waste products can be asserted: (i) the need to use additional additives

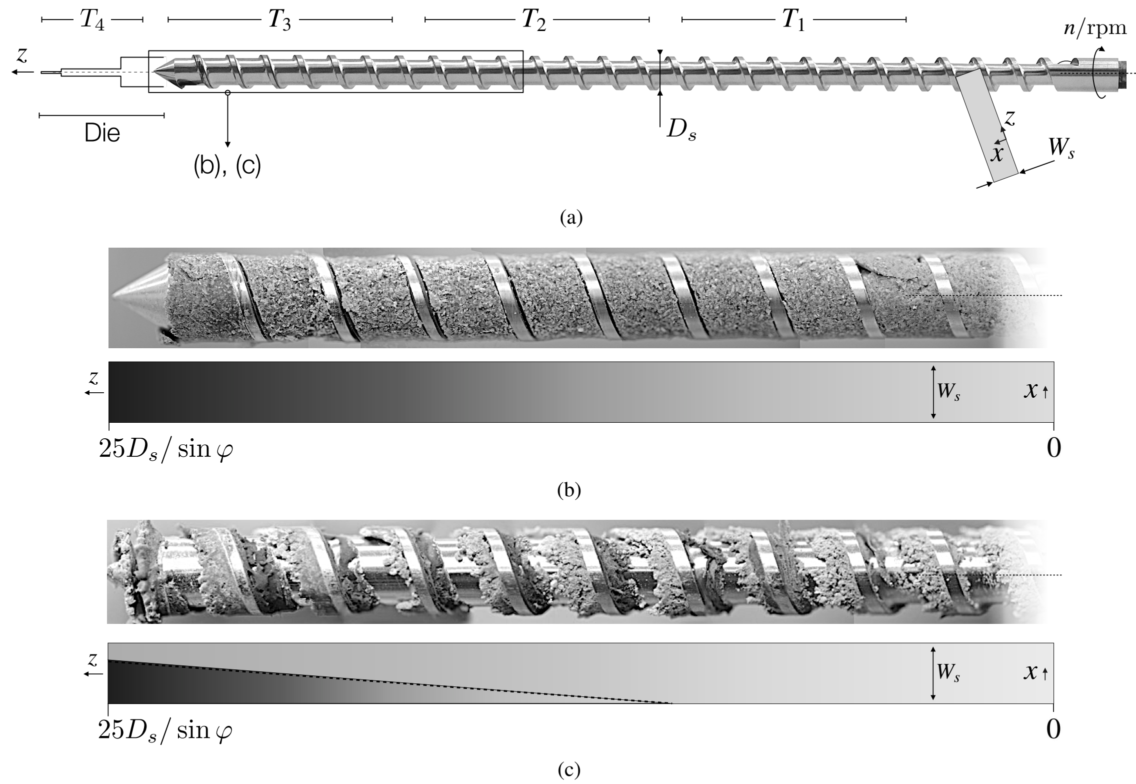


Figure 2. Qualitative overview of two cases of in-situ flow behaviors in an unwrapped screw channel of a compression screw with a compression ratio of 2:1, (a): (b) dispersed plug flow of a oat protein and (c) bifurcating unwrapped screw flow showing the onset of secondary flows with the growth of the melt pool. A moisture content of 40wt% and 45wt%, respectively, was used. The following notations are used in the figure: D_s is the diameter of the barrel, φ is the helix angle, W_s is the width of the screw channel and $T_{1,2,3,4}$ are the extruder heating zones.

in the mixture in order to improve the extrudate's properties towards potential sensory and texture design and to that end (ii) understanding the effect of the thermo-rheological history on the properties of the extrudate. Therefore, in this framework we investigate in this publication the compositional effects of the inline extrusion rheological and microstructural characteristics of novel waste - based high protein content vegetable extrudates.

EXPERIMENTAL

Novel high protein content mixtures based on agro-industrial waste and additional constituents were investigated. The feed composition of the biopolymeric mixtures are shown in Table 1. The compositions were optimized according to processing parameters during pre-

Table 1. The investigated extrusion feed compositions in wt%, where TPM stands for ternary protein mixture. WF for wheat flour, OP for oat protein, PP for potato protein, PS for potato starch and PF for potato fibers.

Composition	WF	OP	PP	PS	PF
TPM	16.7	16.7	16.7	-	-
TPM + Starch	7.0	9.0	9.0	25.0	-
TPM + Fibers	7.0	9.0	9.0	-	25.0
TPM + Starch + Fibers	7.0	9.0	9.0	12.5	12.5

trials. TPM consisted of wheat flour (WF), oat protein (OP) and potato protein (PP). The composition with TPM + Starch had potato starch (PS) which acts as a binding agent. Potato fibers (PF) were added to improve the stability of the extrudates to temperature and shear rates. The processing window was chosen from pre-trials which ensured smooth flow of biopolymeric melt during the extrusion process.

A single screw Brabender 19/25D (barrel diameter of 25 mm and barrel length of 25×19) extruder was used for extrusion cooking and simultaneous inline rheological characterization of biopolymeric mixtures with varying feed composition and moisture contents. The extrusion system comprises four temperature zones, three in the barrel and one corresponding to the extrusion die. The following temperature profile was applied in all the experiments reported in this publication: $90/100/110/120°$ C for zone 1 / zone 2 / zone 3 / zone 4 respectively, where zone 1 corresponds to the feeding region and zone 4 to the die. A compression screw (compression ration 2:1) and a custom built extrusion die complete the extrusion setup. The custom die, having a with and height of $W = 20$ mm and $H = 2$ mm, was equipped with Terwin 2000 melt pressure sensors (max. pressure of 700 bar) connected to a custom built data acquisition system comprising a NI data acquisition board and a LabVIEW code for data pre-processing, including oversampling.[9] Considering the (relatively) high shear rates attained during the extrusion cooking only the power law region of the viscosity function was considered and this the experimental data were fitted with the Power law model, defined by[10]

$$\tau_w = K\dot{\gamma}_a{}^n \tag{1}$$

where τ is the (wall) shear stress computed as

$$\tau_w = \frac{\Delta p}{L}\frac{H}{2} \tag{2}$$

using the pressure loss Δp over the distance L, and $\dot{\gamma}_a$ is the apparent shear rate computed as

$$\dot{\gamma}_a = \frac{6Q}{WH^2} \tag{3}$$

where Q is the (volumetric) flow rate. The consistency index K and the flow behavior index n constitute the parameters of the power law model. The pressure transducer was used to measure pressure difference (Δp) for different screw speeds and steady state values were converted to inline viscosity readings as $\eta_a = \tau_w/\dot{\gamma}_a$.

Microstructural investigations in the extrudate output for observing the shape and size distribution of proteins was the objective of

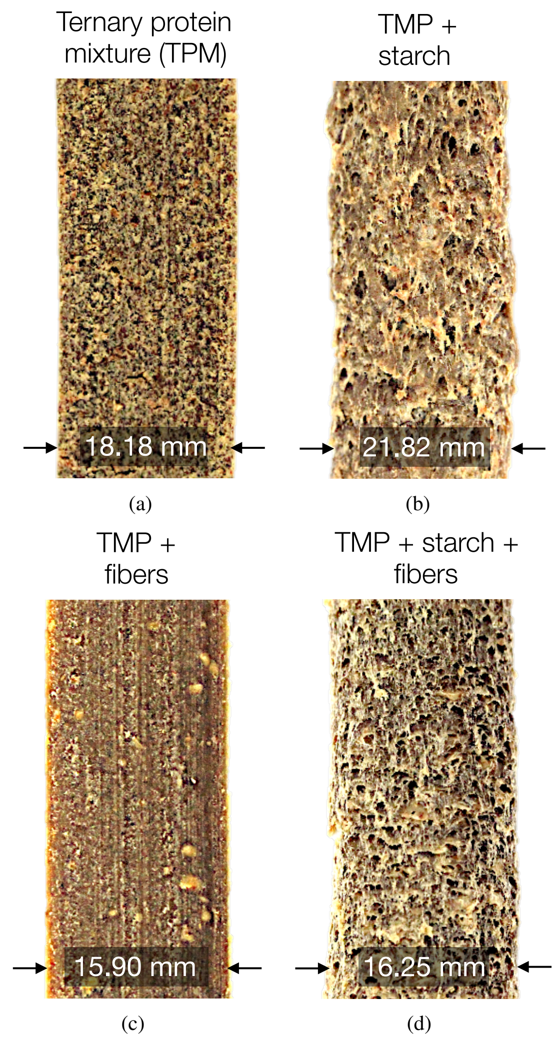


Figure 3. (color online) Optical visualizations of the extrudate output for (a) ternary protein mixture (TPM), (b) TPM + starch, (c) TPM + fibers and (d) TPM + starch and fibers, with a moisture content of 45wt%. The apparent shear rate during extrusion was $\dot{\gamma}_a = 108$ s^{-1}.

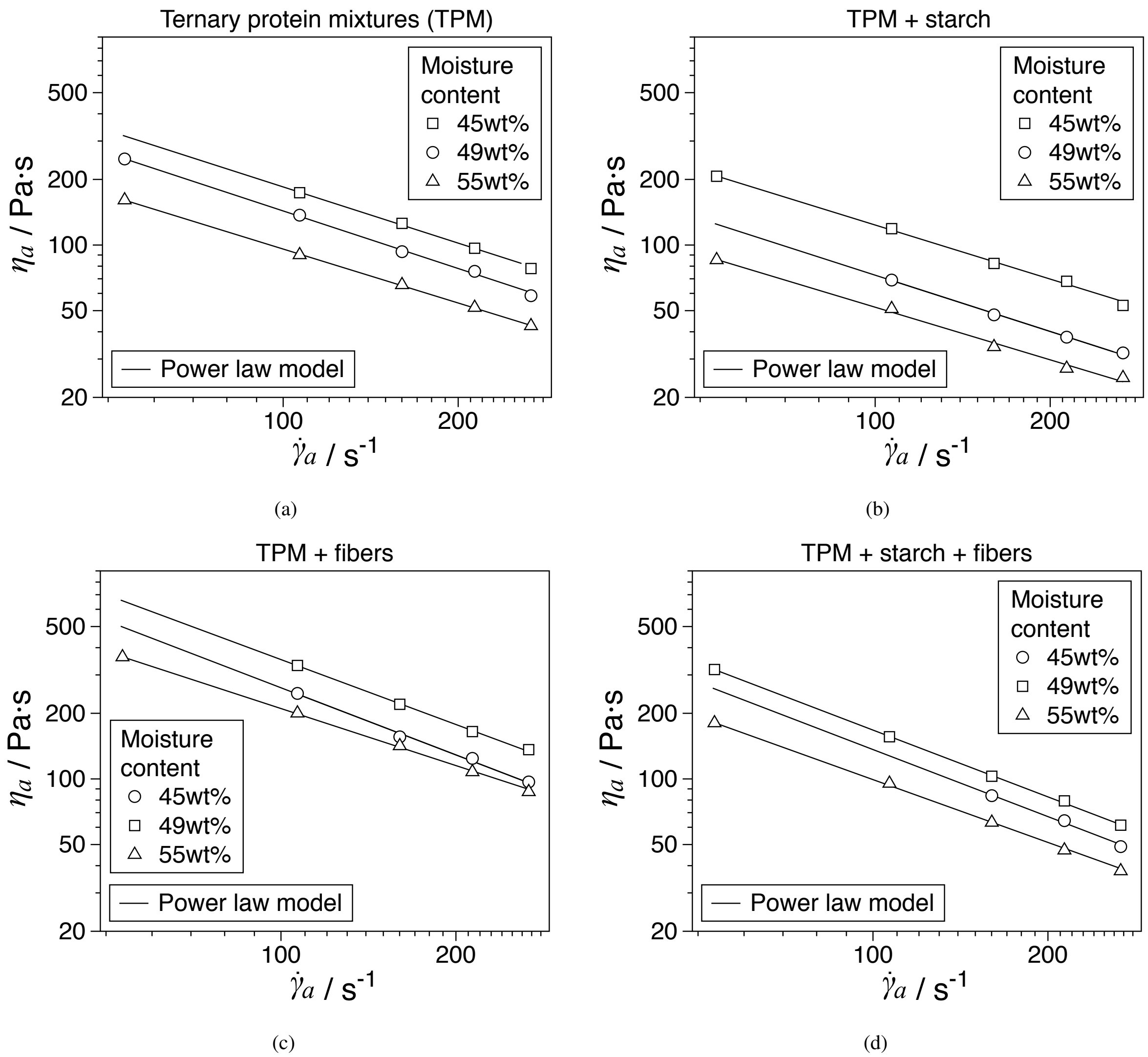


Figure 4. The effect of moisture content on the shear apparent viscosity of (a) ternary protein mixture (TPM), (b) TPM + starch, (c) TPM + fibers and (d) TPM + starch and fibers. The data was fitted using the power law model, Eq. (1).

using light microscope. Sample preparation was performed with cryo-cutting of frozen extrudates and fixating the cut samples for 2 hours. Iodine and light green was used for staining starch and proteins respectively. Additionally, the thermal transition temperatures for the feed powders were evaluated using DSC measurements, however the results are not reported in this publication. Overall, second order glass transitions were observed for all the samples analyzed showing the amorphous nature of foods.

RESULTS AND DISCUSSION

Representative optical visualizations of the extrudate output for the feed compositions investigated are presented in Figure 3, highlighting the variety of textures that can be obtained. It should be noted that while attempting to improve the water stability of TPM extrudates, Figure 3(a), by the addition of starch and fibers to the base composition the texture is significantly altered due to changing rheological properties

Experimental apparent viscosity function extrusion inline determinations together with

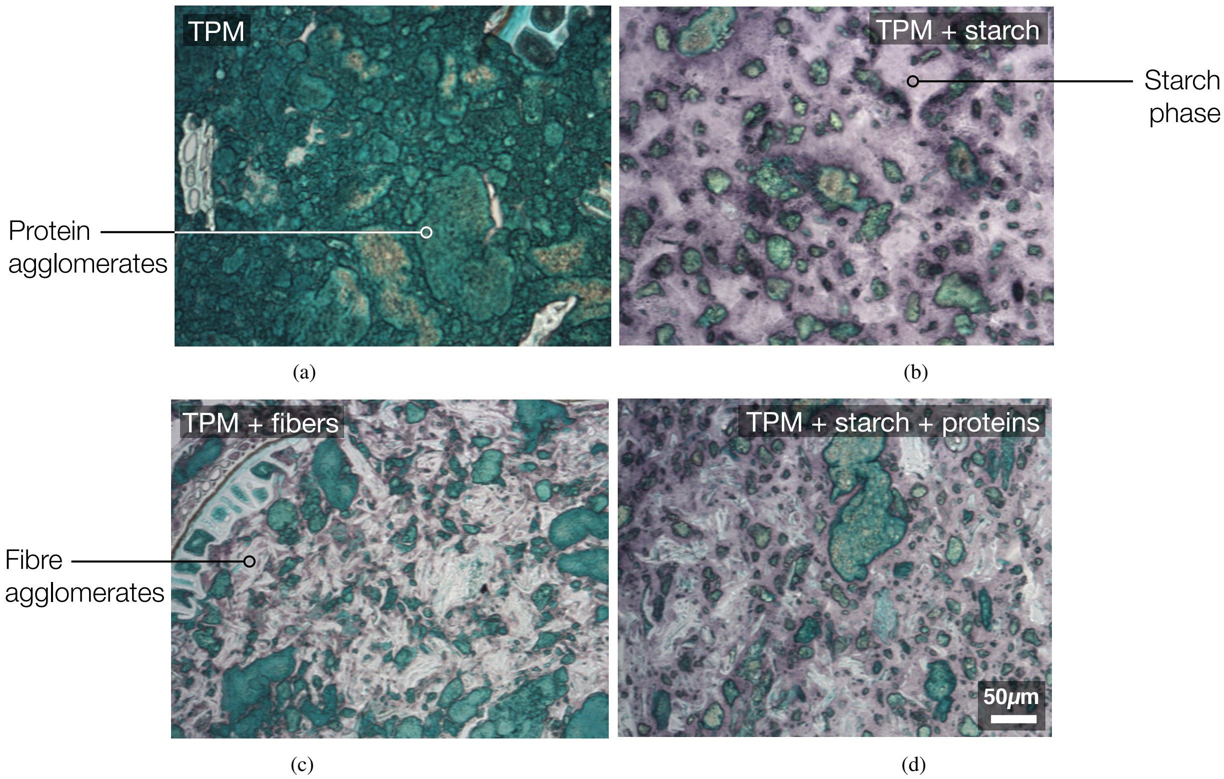
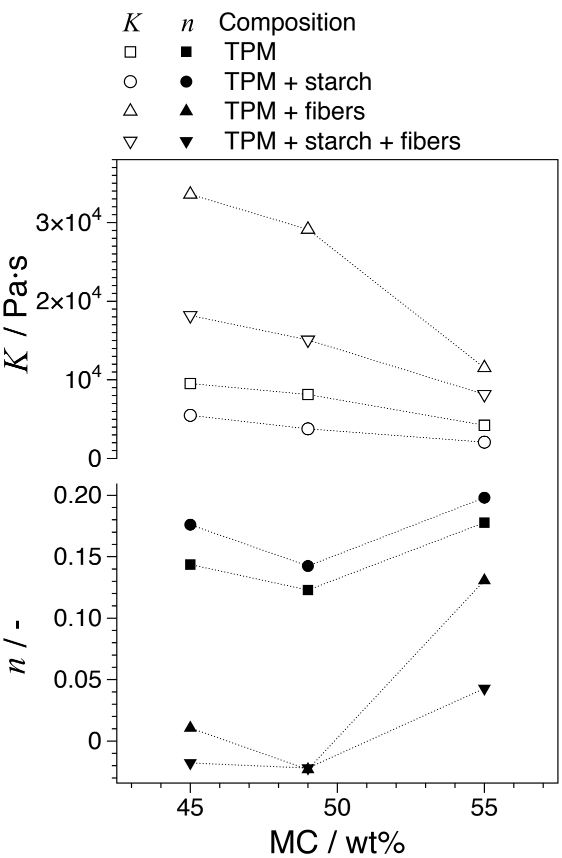


Figure 5. (color online) Microscopy optical visualizations of the extrudate output for (a) ternary protein mixture (TPM), (b) TPM + starch, (c) TPM + fibers and (d) TPM + starch and fibers.

power law model fits are presented in Figure 4 for the feed compositions investigated, with the moisture content as parameter. The continuity of extrudate output was greatly influenced by moisture content. The results appear to validate the hypothesis of an exclusively shear thinning behavior in the (apparent) shear rate range accessible experimentally independent of the feed protein composition and the moisture content used. Differences in the magnitude of the viscosity as well as the shear thinning behavior are however observable.

Qualitative size and shape distributions of the food composition from light microscopic observations are shown in Figure 5. When considering ternary protein mixtures (TPM) are dominated by protein agglomerates, Figure 5(a). Microstructural observations of the composition containing TMP and starch shows that the starch forms a continuous phase after the gelatinization process,[11,12] Figure 5(b). The composition containing TPM and fibers indicates the presence fibre agglomerates, containing possibly entangled fibers, Figure 5(c). Biopolymeric mixtures of TPM with starch and fibers can be interpreted as starch forming a matrix of continuous phase incorporating protein and fiber agglomerates, Figure 5(d).

To summarize the influence of the feed compositional effects on the rheological characteristics rheological behavior observed, the variation of the power law model parameters, the so-called flow consistency index K and flow behavior (power) index n in Eq. (1) are presented in Figure 6. With respect to the influence of the feed compositions on the magnitude of the (apparent) viscosity, i.e. K, it is observed that with the addition of starch, the continuous starch phase formed through gelatinization, see Figure 5(b),(d), has a reducing effect on the flow consistency index K. This can be seen when comparing TPM with TPM + starch and TPM + fibers and TPM + starch + fibers. The addition of fibers has an increas-



(a)

Figure 6. Variation of the power law model parameters, the so-called flow consistency index *K* and flow behavior (power) index *n*, with the moisture content for the feed compositions investigated.

ing effect on *K* acting as filler as the all *K* values reported for compositions with fibers are higher than those in the absence of fibers. As expected, the moisture content has a reducing effect on the magnitude of the apparent viscosity, i.e. *K*. However, this effect is more significant for compositions containing fibers with the highest decrease in *K* with MC being recorded for the TPM + fibers composition. This could explained as the cellulose fibers absorb moisture making them more flexible and thus are more prone to moisture effects. With respect to the influence of the feed compositions on the shear thinning behavior, i.e. *n*, it is observed that with the addition of starch a negligible in *n* values are recorded. However, comparably lower values of *n*, i.e. compositions with fibers have more pronounced shear thinning. This could be attributed to a easier alignment of the cellulose fibers in the flow direction.

SUMMARY AND COMCLUSIONS

The viscosities determined inline during the extrusion of novel vegetable high content protein mixtures exhibited a shear thinning behavior in the range of shear rates investigated an thus good power law model correlation. Microstructural investigations revealed existence and distribution of protein and fiber agglomerates as well as the existence of a continuous starch phase when present in the feed composition. The compositional effects and their dynamics during processing were emphasized by the rheologicel behavior recorded, in terms of the parameters of the power law model.

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