



Research Article

**Structural characteristics and functional properties of fiber-rich by-products of white cabbage modified by high-energy wet media milling**

Isis von Ulardt<sup>1</sup>✉, Marlen Springer<sup>1</sup>, Rafael Valbuena<sup>1</sup>

<sup>1</sup> Institute of Agricultural and Urban Ecological Projects affiliated to Humboldt-Universität zu Berlin (IASP), Philippstraße 13 / Haus 16 10115 Berlin, Germany

**Abstract**

The recovery of residues and by-products of the food industry plays an important role in terms of sustainable management. For this reason, the aim of this study was to analyse the effect of wet milling parameters on dietary fiber concentrates of white cabbage by products or, more precisely, the stalks of cabbage. The input of hydraulic shear-energy during wet milling process leads to a partial modification of the structure of fiber components to obtain compounds with high water- and oil-binding properties. Furthermore, the wet milling parameters affect the functional properties of the fiber concentrates. A mathematical model was developed which relates the functional properties to the parameters of the colloid mill such as slurry concentration, milling time, agitation speed and particle size distribution. A slurry of the grounded material is forced into the milling gap. Grinding is autogenous as a result of collisions between rotating particles. All of the material in the process stream is being grounded finer than the gap setting and grinding can be optimized by adjusting mill operating parameters. The identification of the relations between milling parameters and functional properties is necessary in order to comprehend the processing characteristics of the material in the context of fiber enriched food products manufacturing.

**Keywords:** white cabbage, residues, water-binding capacity, oil-binding capacity, colloid mill

**Abbreviations:**

DF – dietary fiber                                  DM – dry matter                                  WBC – water-binding capacity  
DFC – dietary fiber concentrates                  OBC – oil-binding capacity

✉ Corresponding author: Isis von Ulardt, Scientific Assistant, Institute of Agricultural and Urban Ecological Projects affiliated to Humboldt-Universität zu Berlin (IASP), Philippstraße 13 / Haus 16 10115 Berlin, Germany, tel: +49 (0)30 2093 9062, E-mail: [isis.von.ulardt@iasp.hu-berlin.de](mailto:isis.von.ulardt@iasp.hu-berlin.de)

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## Introduction

The intensification of food production in developed countries led to large waste streams of by-products. The inedible fraction of fresh cabbages and other brassicas based upon literature values are approximately 20 % and can be attributed to unavoidable wastes out of industrial processing (Laurentiis et al. 2018). The trimmings, like stalks, are rich in cell wall materials and their high amount of dietary fiber (DF) enables their usage in modeling new natural ingredients for the food industry.

DF are food components well-known for their beneficial effects on human health (EFSA 2010). The most widely accepted classification for DF is the differentiation of dietary components based on their solubility. (Dhingra et al. 2012). The term 'dietary fiber concentrates' (DFC) can be used for a product which major component is DF, but which does not exclude the presence of other components, such as digestible carbohydrates, protein, lipids, minerals and a small amount of water (Garcia-Amezquita et al. 2017).

The physicochemical properties of DFC are an issue for their functionality like characteristic chemistry, dimensions, surface properties and surface charge and can be affected by chemical, enzymatic, mechanical, thermal or thermo-mechanical treatments (Guillon and Champ 2000). One of the most common functional properties of DFC are water- and oil-binding capacities (WBC and OBC) and the bulk density (Garcia-Amezquita et al. 2017). Physical modification of DFC can set out the conditions that are necessary to apply them into food products. One possibility for mechanical modification are wet milling processes like colloid milling. A colloid mill is mainly constructed of a stator and a rotor. The space between these two elements creates a gap for material passage. When the materials pass through, the materials attaching on the rotating surface will have a maximum speed, while the materials on the stator relative to the rotor will be abiding. The result is a high velocity gradient, which leads to grinding by the strong shear (Chen et al., 2018).

Zhu et al. (2014) reported that ultrafine grinding of buckwheat hull insoluble DF could effectively pulverize the fiber particles to submicron scale. As particle size decreased, the hydration properties

(e.g. WBC) were significantly decreased. Jongarootaprangsee et al. (2007) examined the effects of drying temperature and particle size on hydration properties of DF powder from lime and cabbage outer leaves. The particle size of lime residues did not affect the WBC whereas the particle size of cabbage outer leaves significantly decreased the WBC of their fiber powder. Conversely, Raghavendra et al. (2006) described that the reduction in the particle size of coconut residue DFC from 1127 to 550  $\mu\text{m}$  resulted in increased hydration properties; beyond 550  $\mu\text{m}$ , the hydration properties were reported to decrease with decreases in particle size. The OBC was found to increase with decrease in particle size. Furthermore, Zheng and Li (2018) pictured, that the WBC of coconut (*Cocos nucifera* L) cake DF increased when the particle size was reduced from 250 to 167  $\mu\text{m}$ , while the OHC decreased with decreasing particle size.

It can therefore be concluded that the effect of a grinding operation largely depends upon the food material nature on one hand, and the applied technology, the distribution and intensity of the applied stress by the grinding tool on the other hand. Wet grinding can influence the hydration properties of DF and DFC positively, in particular the kinetics of water uptake. As outcome of the increase of surface area, the fibers hydrate more rapidly. However, the reduction of particle size can lead to an adverse change and collapse of the porous structure resulting in a reduction of (capillary) water uptake (Guillon and Champ 2000).

For this reason, the aim of this study was to analyse the effect of different milling parameters on DFC of white cabbage by-products to improve their functional properties.

## Materials and Methods

**Samples collection.** Stalks of white cabbage *Brassica oleracea* var. *capitata* (2018 crop year) were selected from manufacturing process of pickled cabbage obtained from a local producer in the federal state of Brandenburg, Germany and were cut into pieces (approx. 3x3 cm). Samples of 500 g were vacuum packed in light and air tight laminated plastic bags and kept at  $-18^{\circ}\text{C}$  until analysis.

**Obtaining dietary fiber concentrates.** Obtaining DFC was done with a method described elsewhere (Kunzek et al. 2002). The cut stalks of white

cabbage were pre-comminuted with a cutting mill, washed with distilled water and then pressed at 4.8 bar water pressure by means of a rubber bladder and filter cloth (Fa. Paul Arauner GmbH & Co. KG, Germany). The compressed mash residue was washed with distilled water, blanched in citric acid solution (1.2 %), homogenized using Micra disperser and a shredder attachment (16,500 rpm for 5 minutes). Afterwards the shredded material was wet-sieved manually using a sieving tower (Model AS 200 Retsch, Germany). The wet sieving was carried out with distilled water on a 500 µm sieve with 20 µm sieve underneath until the passage had a conductivity of less than 200 µS/cm.

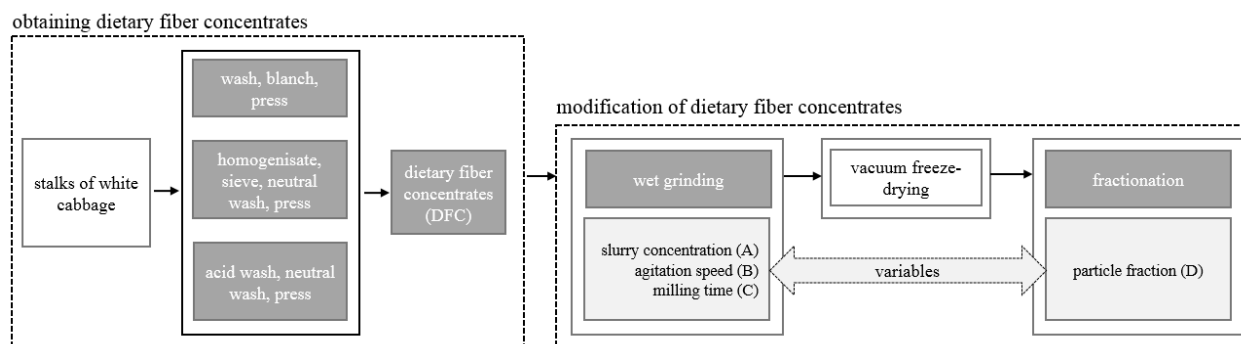
**Modification of dietary fiber concentrates.** The experimental setup of obtaining and modification of DFC is sketched in Figure 1.

**Wet milling.** Sizes of a 0.78 % and 1.04 % w/w DFC suspension in deionized water were reduced using a colloid mill (IKA Magic Lab, Staufen, Germany) and milled under following operation parameters: After 5 and 10 min through the mill and an agitation speed of 14,600 rpm and 20,000 rpm, a sample of each suspension was taken. Grounded fiber concentrates had high water contents. Removal of this water was performed using a

centrifuge (Fa. SIGMA, 6-16 KS; Max. 20,335 RZB) with a rotation rate of 11,200 rpm for 10 min, and the samples were then stored frozen (-18°C).

**Freeze-drying.** To build a porous material out of the colloid milled slurry, the samples were portioned in stainless steel dishes and deep frozen (-18°C) before freeze drying (Christ alpha 1-4, Germany) for about 24 h. The set temperature of the ice condenser was -55°C and the set pressure was adjusted to 0.05 mbar.

**Dry grinding and fractionating.** The freeze-dried samples were grounded in a centrifugal mill (10,000 rpm, model PULVERISETTE 14, Fritsch, Germany) fitted with a 0.5 mm screen to break the freeze-dried agglomerates into smaller units. Grounded samples were separated according to particle size using a sieve shaker (AS 200, Retsch, Germany). Mesh size of sieves (Fa. Retsch) was 20, 200 and 400 µm. Each sample was placed on the top sieve with the largest mesh width and shaken for 10 min at an amplitude setting of 60. The residue of each sieve was kept separately in light and air-tight plastic containers.



**Figure 1.** Obtaining and modification of dietary fiber concentrates.

**Dry matter.** Dry matter (DM) was determined gravimetrically after drying at 105 °C for 24 h.

**Water-binding capacity.** The water-binding capacity (WBC) was determined according to a method described by Robertson et al. (2000). Distilled water (20 ml) was transferred into 50 ml centrifuge tubes containing 200 mg of sample. The mixture was stirred for 5 min and left at room

temperature for 10 min. After centrifugation at 5,000 rpm for 15 min, the excess supernatant was removed and the residue hydrated weight was recorded. WBC was determined as (w/w) deionized water per DM.

**Oil-binding capacity.** Oil-binding capacity (OBC) was measured using a method described by Elleuch et al. (2008). 200 mg of DFC were added to 10 ml

of sunflower oil in a 50 ml centrifuge tube. The content was stirred for 5 min prior centrifugation at 1,500 rpm for 30 min. The free oil was decanted and absorbed oil was determined. OHC was calculated as (w/w) sunflower oil per DM.

**Bulk density.** The bulk density of the dry samples was determined outlined elsewhere (Kaur and Singh 2005). The dry samples were gently filled into 50 ml graduated cylinders, previously tared. The bottom of the cylinder was gently tapped on a laboratory bench several times until there was no further diminution of the sample level after filling to the 5 ml mark. Bulk density was calculated as weight of sample per unit volume of sample (w/v) per DM.

**Statistical analysis.** Data analysis and optimization were done using Two Level Factorial Design. It creates an experiment that includes all possible

combinations of the factor levels. In this design, all factors are being treated numerically. Table 1 represents the levels of independent variables used in the experimental design. Investigated responses (dependent parameters) such as WHC, OHC and bulk density were considered. In this model 16 experiments in triplicate (48 runs) to 2FI full Factorial design were used. The statistical software package Design Expert 12.0 (Stat-Ease, Minneapolis, MN) was used for regression analysis of the data and estimation of regression equation coefficients. Significant terms in the model for each response were found by analysis of variance (ANOVA) and were employed to determine the regression coefficients and statistical significance of the model terms.

**Table 1.** Experimental design of DFC modification.

Factor	Name	Units	Type	Minimum	Maximum	Levels
A	slurry concentration	g/g	Numeric	0.78	1.04	2
B	agitation speed	rpm	Numeric	14,600	20,000	2
C	milling time	min	Numeric	5	10	2
D	particle fraction	µm	Numeric	20-200	200-400	2

## Results and Discussion

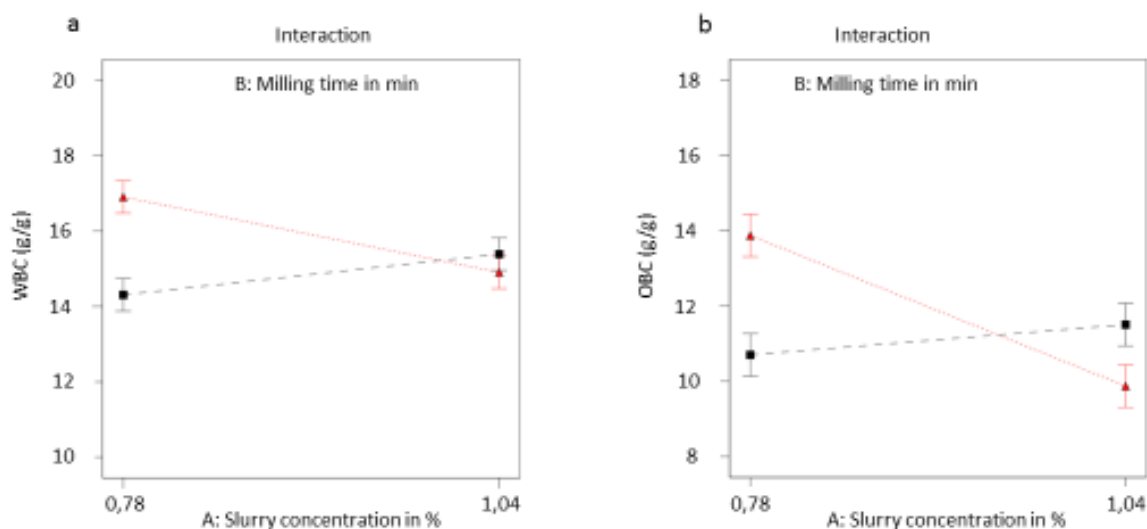
**Analysis of variance, fitting the model.** In order to study the effects of the four independent variables namely (A) ‘slurry concentration’, (B) ‘milling time’, (C) ‘agitation speed’ and (D) ‘particle fraction’ on the dependent variables WBC, OHC and bulk density, a two level factorial design methodology was applied. The Model F-values of all dependent variables imply significance of models ( $p < 0.0001$ ) at the designated conditions. For WHC B, C and their interactions AB, ABD, BCD had significant effects ( $p < 0.001$ ). For OHC the factors B, C, D, and their interactions AB, AC, ABC had significant effects ( $p < 0.002$ ). Although the main effect of A was not significant, it was also included to achieve hierarchic models. The factors that had significant effects on the bulk density were A, B, C, D and the interactions of AC, AD, BC, CD, ABC, and ABCD ( $p < 0.0001$ ). In all cases, the Predicted  $R^2$  was in reasonable agreement with the Adjusted  $R^2$ ; i. e. the difference is less than 0.2. The

measured Adeq Precision indicated in all cases an adequate signal in relation to the signal to noise ratio.

**Water-binding capacity.** The WBC of DFC ranged between 12.4 and 17.6 g/g, which is in accordance with data typically found for fruit and vegetable fibers in literature (Elleuch et al. 2011). The interaction effects which were considered to be significant by ANOVA were studied. The normal probability plot and Pareto plot showed that the most significant factor with positive effects was the ‘particle fraction’ (D) followed by the ‘milling time’ (B). All samples of the series revealed higher WBC at higher ‘particle fraction’ of agglomerates. The WBC refers to the amount of water that remains bounded within the fiber structure after presence of external force such as centrifugation. Gupta and Premavalli (2016) also reported that the WBC of vegetable fibers were higher at higher ‘particle

fraction' as the particles are less firmly packed than finer ones. The 'agitation speed' (C), the interaction between 'concentration and milling time' (AB) as well as the interaction between 'concentration, time and particle fraction' (ABD) had significant negative effects on WBC. The interaction between 'concentration and time' (AB) showed significant

effects on WBC when 'particle fraction' was on its high level for both levels of 'agitation speed' as shown in Figure 2 a. Increase in initial 'slurry concentration' increased the WBC of the DFC at low 'milling time', while increase in 'slurry concentration' decreased the WBC at high 'milling time'.

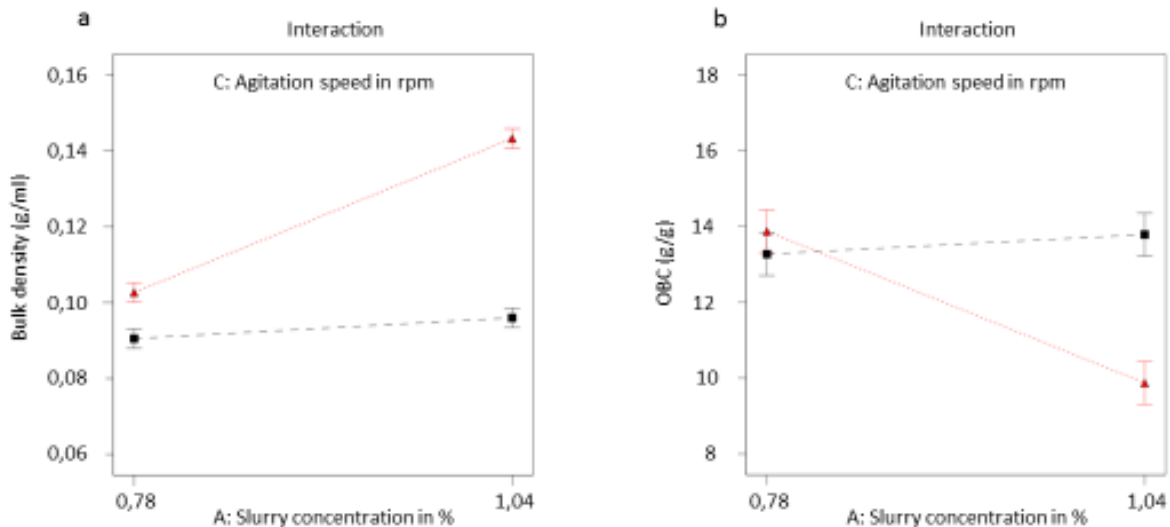


**Figure 2.** Interaction plot for (a) water and (b) oil-binding capacity of dietary fiber concentrates produced with different slurry concentrations and milling times ( $\blacktriangle$ =10 min,  $\blacksquare$ =5 min) and at agitation speed and particle fraction on + level. ( $\pm$ LSD)

**Oil-binding capacity.** The OBC of samples tested amounted to a value between 9.4 g/g and 15.0 g/g. The interaction effects considered significant ( $p < 0.001$ ) by ANOVA were studied. The normal probability plot and Pareto plot showed that the most significant factor with negative effects is the 'agitation speed' (C) followed by the interaction between 'concentration and time' (AB), and the interaction between 'concentration and agitation speed' (AC). The higher these factors, the lower the OBC of the samples. In addition, 'particle fraction' had significant positive effects on OBC. The oil-binding capacity is related to the quality of the surface and the density or thickness of particles, so that those particles with the greatest surface area theoretically present a greater capacity to adsorb and bind components of an oily nature (Lopez et al. 1998). The interaction between 'concentration, time and agitation speed', and the factor 'time' were important but less significant ( $>$  Bonferroni limit).

The interaction between 'concentration and time' (AB) showed no significant effect when 'agitation speed' was at its - level. When 'agitation speed' was on its + level, the change in 'slurry concentration' had an effect on the OBC at both high and low 'milling time'. Increase in 'slurry concentration' increased the OBC of the DFC at low 'milling time', while increase in initial 'slurry concentration' decreased the OBC at high 'milling time'. Figure 2 b illustrates the significance of interactions of AB. The deviation from the parallel of the lines is related to the degree of interaction. In other words, the effect of one factor depends on the level of the other. The interaction between 'concentration and agitation speed' (AC) showed no significant effect when 'milling time' was at its - level, for both levels of 'particle fraction'. When 'milling time' was on its high level, the OBC was significantly different at high 'slurry concentration' (Fig. 3 b).





**Figure 3.** Interaction plots for (a) bulk density and (b) oil-binding capacity of dietary fiber concentrates produced with different slurry concentrations and agitation speed ( $\blacktriangle$ =20,000 rpm,  $\blacksquare$ =14,600 rpm) at milling time and particle fraction on + level. ( $\pm$ LSD)

**Bulk density.** The determined bulk density ranged from 0.07 to 0.14 g/ml. ANOVA revealed the highest significant positive effects ( $p < 0.001$ ) for the factors ‘slurry concentration’ (A), ‘agitation speed’ (C), their interaction (AC) and ‘particle fraction’ (D) with regard to the bulk density. The effects of interaction AC on bulk density is shown in Figure 3 a. The highest significant negative main effects on bulk density had the interaction between BC and the factor ‘milling time’. The interaction between BC showed significant effects by increasing ‘agitation speed’ for both levels of ‘milling time’ and ‘particle fraction’. Typically, a decrease in fiber ‘particle fraction’ is associated with an increase in bulk density (Sangnark and Noomhorm 2003). However, the method applied for these trials directed towards an alternative conclusion. Due to previous wet milling prior to grinding and fractionation, the agglomerates are made of tightly packed particles with irregular and frayed surfaces. As a result, even the bigger fiber particles can join together more densely.

## Conclusions

The present study shows that several parameters during mechanical pre-treatment affected the structural characteristics and physicochemical

properties of fiber-rich by-products of white cabbage. A high level of ‘particle fraction’ (+D) of agglomerates turned out to be one of the key factors to increase WBC and OBC. The high bulk density at high level ‘particle fraction’ (+D) suggests that the agglomerates are made of tightly packed particles with irregular and frayed surfaces due to a wet milling process before drying. The bulk density was found to be inversely proportional to the OBC when DFC were produced at high level ‘agitation speed’, ‘milling time’ and ‘particle fraction’. The highest values for both WBC and OBC were measured using the following factor settings: low level ‘slurry concentration’ (-A), high level ‘milling time’ (+B), high level ‘agitation speed’ (+C) and high level ‘particle fraction’ (+D). High water- and oil-binding capacity opens up the possibility of using the fibers as ingredients in food products. DFC with high OBC provides the stabilization of high fat food products and emulsions. Dietary fibers with high WBC can be applied as functional ingredients to avoid syneresis and modify the viscosity and texture of some formulated foods (Elleuch et al. 2011). The identification of the relations between processing and functionality is necessary to know the processing characteristics of the material in the context of the manufacture of fiber enriched food products.

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