O4-2 **Microencapsulation of polyphenol extracts derived from apple pomace for utilization as food additives**

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INTRODUCTION AND OBJECTIVES

Polyphenols are secondary plant metabolites occurring naturally in fruits and vegetables. Many studies could show potential health benefits associated with these bio-active compounds (Middleton 2000). A possibility to increase polyphenol ingestion is the selective enrichment of foods with plant extracts of high polyphenol content. This application is limited by the extract's taste, which is characterized by the astringency and bitterness of the polyphenols and by the specific taste due to the raw material (Lesschaeve 2005). Thus, the aim of this work was masking the taste of polyphenol extracts by microencapsulation. Extracts were derived from apple pomace as a cheap raw material with high polyphenol content. By screening various wall materials, promising materials and material combinations were identified, and the encapsulation process was optimized for them.

MATERIALS AND METHODS

Materials

Pectins and apple pomace were provided by Herbstreith & Fox KG. Lupine and pisane were from Georg Breuer GmbH, modified starches from National Starch. All other encapsulation materials were provided by Rudolf Wild GmbH & Co. KG.

Polyphenol extraction

Dried apple pomace was milled to 2 mm and polyphenols were extracted under optimized conditions identified in preliminary experiments (1 h, 47 °C, 48% (v/v) aqueous isopropanole, 200 g pomace/ L. Isopropanole was removed by rotary evaporation (220 mPa, 65 °C, 18 min).

Screening

Spray drying: 20 materials of different groups (proteins, starches, celluloses, gums) were investigated. Extract and wall material were suspended in the ratio 1:2 in water, these liquid formulations were analyzed for its sensory characteristics and interactions between polyphenols and wall material. The 5 most promising wall materials were selected and mixed in 24 different combinations. These combinations were then spray dried (ratio extract to wall material 1:2, 10% dm, 180/ 80 °C). Powders were analyzed for its sensory characteristics, retention of polyphenols and dry mass.

Combined precipitation and spray drying: cold extrusion approaches with 7 materials were conducted (2 high, 2 low esterified, 2 amidated pectins, sodium-alginate). Additionally, encapsulation of the extract by rennet gelation of skim milk (based on Heidebach 2009) was investigated. Ratio of extract to wall material was 1:2 in all cases and extract was mixed with the wall materials prior to precipitation. 2% of each wall material was solved in distilled water. Solutions of sodium-alginate, amidated and low esterified pectins were precipitated in 0.13% calcium-lactate, solutions of highly esterified pectins were atomized into 10% saccharose, adjusted to pH 2.6 with lactic acid. After precipitation maltodextrin (50 % of the dry mass) was added. In addition to these approaches one multilayer capsule consisting of two oppositely charged materials (pisane$^{(+)}$, highly esterified pectin$^{(-)}$) and corn maltodextrin (27.5:22.5:50) (based on Gharsallaoui 2010), was produced. All samples were spray dried at 180/ 80°C and analyzed the same way as the material combinations above.

Process optimization

Encapsulation was optimized for the 3 most promising wall materials and wall material combinations (ML: multilayer, P: lupine, pisane, S: modified starch, E1450). Following parameters were varied: ratio wall material 1 to wall material 2 (only for P), ratio extract to wall material, dry mass of wall material, dry mass of maltodextrin (only for ML), inlet temperature, outlet temperature. At each level one parameter was varied. The next levels were conducted with the optimized parameters of the previous levels. All resulting powders were analyzed for its sensory characteristics, polyphenol retention and dry mass.

Sensory evaluation

Relevant gustatory perceptions were identified by a simple descriptive test according to DIN 10964 with native apple extract. For liquid formulations, process optimization of ML and P and comparison of the optimized powders ML, P and S detection thresholds were identified according to DIN 10959. For each formulation 7 samples of increasing polyphenol content (0 to 30 mg/ 200 g matrix) were tested by 5 trained panelists. Spray dried material combinations and powders of process optimization S were tested by the consensus profile method according to DIN 10967 by 12 trained panelists with samples of 42 mg total phenolic content/ 200 g matrix. For all powders the matrix used was yoghurt, liquid formulations were tested in MCT-oil with 4% Span 60.

Determination of polyphenols by HPLC

Extraction of powders was performed using a modified pressurized liquid extraction method (based on Saénz 2009). After diluting the extracts and liquid formulations with distilled water, an automated solid phase extraction was carried out using polyamide solid phase cartridges

Phenolic compounds were identified by RP-HPLC-DAD-MS^n and quantified in comparison to authentic standards (Wollseifen 2010). Interactions between polyphenols and wall material were identified by centrifugation (13.000 rpm, 10 min) of the liquid formulations and determination of polyphenols in the supernatant. Polyphenol retention was calculated by comparing phenolic content of the extract before and after spray drying.

RESULTS AND DISCUSSION

Single materials showed significant differences in masking *apple taste* and *astringency*. In case of *apple taste* the modified starch E1450, two maltodextrins (from tapioca and corn starch), hydroxypropylmethylcellulose and lupine showed highest masking potential. For *astringency* modified starch, corn maltodextrin and the proteins pisane, lupine and whey protein had best results. Reduced astringency perception by utilization of proteins may be due to strong protein-polyphenol-interactions (48-71%). 24 combinations of pisane, lupine, whey protein, modified starch and corn maltodextrin were spray dried. Evaluation revealed that modified starch and combinations of the proteins lupine and pisane lead to best results in terms of masking the gustatory perceptions. For all powders produced by precipitation as well as for the multilayer powder perception of *astringency* was strongly reduced. *Apple taste* was masked best by multilayer. Thus, process optimization was conducted for multilayer, for modified starch and for combinations of lupine and pisane. Optimized conditions for all three approaches are presented in table 1.

Table 1 : results of process optimization

	1	2	3	4	5	6
ML	---	1:2	3.4	50	180	80
P	0:1	1:4	15	---	180	80
S	---	1:3	20	---	140	60

ML: multilayer, P: proteins, S: modified starch; 1: ratio lupine : pisane, 2: ratio extract : wall material, 3: dry mass of wall material [%], 4: portion of maltodextrin [%], 5 / 6: inlet / outlet temperature [°C]

All optimized powders had high polyphenol retentions (89-98%) and dry masses (95-96%). Comparison of the detection thresholds for the relevant gustatory perceptions showed that both multilayer and pisane encapsulation increased detection thresholds remarkably. *Apple taste* and *astringency* were not detected even at a total phenolic content of 30 mg/ 200 g. In case of modified starch thresholds were lower, *apple taste* was identified at 11 mg/ 200 g, and *astringency* was detected at 23 mg/ 200 g. Thus, powders of the multilayer and protein approaches seem to be most suitable for encapsulation of polyphenols from apple pomace. In case of multilayer optimized powders contained 33.33% extract, encapsulation with pisane had to be conducted with extract-portions of 20% to yield comparable results. However, with wall material dry masses of 15%, spray drying of pisane is more efficient, since for multilayer only dry masses of 3.4% can be realized due to the pectin's high viscosity.

CONCLUSIONS

In this research a screening of various materials and material combinations for encapsulation of polyphenol extract from apple pomace was conducted. By optimization of the encapsulation process, sensory properties of the polyphenol powders could be improved. Utilization of pisane as single wall material as well as combined with pectin (multilayer) is most effective in masking undesired taste of the extract. Microencapsulation of polyphenol extracts seems to be promising for masking their taste and making them applicable for food enrichment.

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