Pol. J. Food Nutr. Sci. 2006, Vol. 15/56, SI 1, pp. 87–90

# CHARACTERISTICS OF ACID WHEY POWDER PARTIALLY DEMINERALISED BY NANOFILTRATION

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Key words: whey powder, acid whey, nanofiltration, demineralisation, neutralisation, physicochemical properties, solubility

Abbreviations: **AW** – acid whey, **AWP** – acid whey powder, **DF** – diafiltration, **SWP** – sweet whey powder, **NF** – nanofiltration, **NEU** – neutralisation

The chemical composition and solubility of acid whey powders obtained from nanofltered and neutralised wheys were studied. The change in the composition and acidity of acid whey powders caused by nanofiltration and neutralisation significantly influenced their solubility. With a decrease in ash content and acidity, the solubility was observed to increase. The correlation was found between ash content ($r$=0.843) and acidity to insolubility index ($r$=0.709). The study showed that the location of neutralisation in the technological process of acid whey powder had a significant influence on its properties. More favourable would be to apply acid whey neutralisation after nanofiltration. Diafiltered acid whey powders were characterised by the best properties, even better than those of the control sweet whey powder.

## INTRODUCTION

Worldwide growing amount of whey is industrially processed into whey powders and other high-quality protein-rich products meant for nutritional use. The food market research reveals an increasing interest in desalted whey products. Whey used for human consumption will increase in value if demineralised [van der Horst *et al.*, 1995]. The field of the application of desalted whey in the food industry is much wider than that of un-fractionated one. Such products are used by the food industry in the production of instant formulas for children (90% desalination), confectionery, bakery and meat products, ice creams, frozen dessert, snack foods and dressing (50–75% desalination) as mainly a skim milk powder substitute because of economic reason [Glover, 1985; Boumba *et al.*, 2001].

Acid whey is a substantial by-product of the tvarog production, which is a very popular fresh cheese in Poland. Whey components possess numerous nutritive and functional properties and thus acid whey should be utilised as an ingredient in a variety of food products. Whey powders used for food production increase their nutritional profile with complete and bio-available amino acids and offer functional benefits to food formulators, such as improving the product's sensory attributes, emulsification and enhancing the moisture retention.

Because of technological problems, the food industry has not fully recognised the potential of acid whey as a food ingredient yet. Until now, a substantial amount of acid whey has been used as a component in animal feeds or even treated as inconvenient waste [Morr, 1990; Morr & Ha, 1991].

Drying acid whey is troublesome due to its high lactic acid content. During spray drying lactic acid proceeds agglomeration, which causes lumping and caking of powder particles. Moreover lactic acid increases whey viscosity and lowers the quality of whey products. Drying acid whey is possible after neutralisation or addition of skim milk or grain products [Bylund, 1995]. The physicochemical properties of whey powder, being a food ingredient, affect the quality of the final product, most notably during storage and distribution [Saltmarch & Labuza, 1980]. The largest structural element of AWP particle is lactose [Mulvihill & Grufferty, 1997]. Depending on lactose form, which can be amorphous or crystalline, AWP can be hygroscopic or non-hygroscopic. Lactose should be crystallised in whey concentrate in order to prevent whey powder caking. The functional properties of AWP may vary depending on the composition, protein solubility, bulk density and particle size distribution. Therefore it seems necessary to collect data about the physicochemical properties of desalted AWP.

The objective of this study was to produce, characterise and compare the physicochemical and functional properties of acid whey powder with those of commercially manufactured sweet whey powder (SWP). It is reasonable to assume that such knowledge can extend potential food applications of dried whey products. The demineralised and deacidified AWP would be available for example for incorporation into other dairy products, such as ice cream and yoghurt [Nguyen *et al.*, 2003].

## MATERIALS AND METHODS

**Materials.** Acid whey powders were produced in a laboratory scale from tvarog acid whey by nanofiltration (NF),

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neutralisation (NEU) and subsequent evaporation and spray-drying. During the experiments, the influence of NF and NEU on AWP was examined. The following NEU variants were used: (1) before NF, (2) after NF and (3) after diafiltration (DF). Acid whey neutralisation was carried out by adding 30% NaOH solution to acid whey in order to imitate its pH to sweet whey.

Cooled to 4°C tvarog acid whey was collected from a local dairy. After defatting and clarifying by centrifugation at 4.500 g whey was pasteurised at 74°C for 60 min and three times concentrated by nanofiltration at a temp. of 18–22°C and under operative pressure of 23–26 bar to 17–18% solids content. The NF retentate obtained was further evaporated in an Anhydro type Lab E evaporator, crystallised and spray dried in a Niro Atomizer spray tower at inlet and outlet temperatures of 180°C and 80–90°C, respectively.

The SWP samples examined in this study originated from Polish dairy. The whey powder samples were assayed immediately after production.

**Methods.** The following analyses were carried out in this research: moisture, protein, lactose, ash and some minerals contents and insolubility index, pH and titratable acidity of 10% whey powder solutions. The analyses were performed with the following methods: moisture content with a standard oven-drying method, lactose content with the Bertrand's method, protein content with the Kjeldahl's method, ash content by incineration in muffle furnace (at 500°C by 4 h), calcium and sodium contents with the method decsribed by Whiteside and Miner [1984], titratable acidity and insolubility index according to IDF standards [IDF, 1981, 1988]. Each analysis was carried out in duplicate.

**Statistical analysis.** The properties of the powders were correlated by calculating a correlation coefficient using statistical functions of Microsoft Excel software.

## RESULTS AND DISCUSSION

The obtained AWP samples were compared to the industrially produced SWP. For the reason of the type of whey, mechanism of curd formation and addition of the neutraliser the main differences in whey powders concerned acidity, ash content and solubility.

The changes of acid whey powders properties produced by the effects of NEU, NF and DF are shown in Table 1.

The water content is a property of major importance in milk powders because of its influence on the shelf life and textural and technological qualities. In this study, moisture content of whey powders varied from 1.3 to 4.8%, with an average of 2.66%. All samples of whey powders were characterised by similar lactose contents (SWP – 76.3%, AWP 68.0-75.1 with an average of 71.06%).

The high ash and lactic acid contents in acid whey significantly limit its industrial processing. Hence acid whey was concentrated by NF, which decreased its ash content and acidity.

The ash content of AWP was by about 40% higher than that of SWP. Acid whey powders produced from neutralised acid whey were characterised by even higher ash content. This feature caused salty taste of powder, which in practice, made its use in food products impossible. Next, whey powder manufactured from only evaporated and dried acid whey was characterised by the highest acidity (pH and °SH – 4.91 and 25.5, respectively). These features suggested including both NEU and NF to technological processing. However NF process did not cause any significant reduction of AWP acidity. This indicated the necessity of including NEU into AWP technological process.

TABLE 1. Physicochemical properties of whey powders.

| Whey powder | Parameter | | | |
|---|---|---|---|---|
| | 10% solution | | Ash content (% of total solids) | Insolubility index ($cm^3$) |
| | pH | Titratable acidity °SH | | |
| SWP | 6.24 | 6.5 | 9.17 | 0.5 |
| AWP | 4.91 | 25.5 | 12.93 | 3.25 |
| AW-NEU | 5.59 | 13.5 | 14.09 | 4.28 |
| AW-NF | 4.61 | 24.8 | 9.27 | 3.00 |
| AW-NEU-NF | 5.48 | 13.3 | 10.84 | 2.18 |
| AW-NF-NEU | 5.84 | 8.2 | 7.55 | 1.67 |
| AW-NF-DF-NEU | 6.28 | 5.4 | 5.18 | 0.40 |

AW-NEU – acid whey powder from neutralised acid whey; AW-NF – acid whey powder from nanofiltered acid whey; AW-NEU-NF – acid whey powder from neutralised and nanofiltered acid whey; AW-NF--NEU – acid whey powder from nanofiltered and neutralised acid whey; AW-NF-DF-NEU – acid whey powder from nanofiltered, diafiltered and neutralised acid whey

The results obtained showed that the location of NEU had a significant influence on AWP properties. As it turned out NEU after NF was a more advantageous variant than before because of lower acidity and ash content in so-produced AWP.

Samples of whey powders from diafiltered acid whey were described by the lowest acidity, even lower than that of SWP. Diafiltration also increased ash content reduction in AWP. The samples of AWP from diafiltered acid whey were characterised by the lowest ash content, that was lower than in SWP (5.18% and 9.17% in total solids, respectively).

For non-neutralised acid whey nanofiltration allowed concentrating and demineralising whey by almost 30% (reduction in ash content from 12.93% to 9.27% in total solids). The results obtained showed that demineralisation by NF is more effective for acid whey at pH 4.50. This means that the mineral permeation process through NF membrane is more effective at lower pH value of raw material. Moreover it turned out that acid whey neutralisation after NF (so neutralisation of NF retentate not whey) was more advantageous because it led to a more effective reduction in the contents of acids (mainly lactic).

Barrantes & Morr [1997] also showed that NF method enables effective removal of both lactic acid and ash from cottage cheese whey. Additionally, besides whey desalination and deacidification, NF concentrates whey to about 20% of total solids [Kelly & Kelly,1995].

In this study, contents of some minerals in whey powders were studied as well. Because of ion-selectivity of NF membrane, sodium was chosen as a representative of monovalent and calcium as that of multivalent ions (Table 2).

Sodium content of the examined AWP samples varied from 2.49 to 14.22 mg/g (Table 2). Obviously the highest sodium content was observed in AWP from neutralised acid whey because of the addition of this element as a neutraliser. As expected, the AWP samples from nanofiltered acid whey were characterised by the lowest sodium content (lower by 75% than in AWP from acid whey). This shows that NF membrane has a great potential to reduce the concentration of monovalent ions. This ability was higher for AWP from acid whey neutralised after NF. Diafiltration process further reduced the sodium content to a value lower (8 mg/g) than that reported for SWP (9.1 mg/g).

Calcium content of AWP samples varied from 10.67 to 19.40 mg/g (Table 2). It showed that the effectiveness to reduce multivalent ions content by NF membrane was significantly less than for the monovalent ones. Also Boumba *et al.* [2001] stated that the level of multivalent ions, such as calcium in whey powders remained almost constant [Boumba *et al.*, 2001].

TABLE 2. Comparison of the contents of selected minerals of whey powders.

| Whey powder | Content (mg/g) | |
|---|---|---|
| | Ca | Na |
| SWP* | 3.90 | 9.10 |
| AWP* | 19.40 | 9.90 |
| AW-NEU | 13.89 | 14.22 |
| AW-NF | 14.15 | 2.49 |
| AW-NEU-NF | 18.78 | 11.29 |
| AW-NF-NEU | 13.38 | 9.59 |
| AW-NF-DF-NEU | 10.67 | 8.08 |

Explanations as in Table 1; * according to Bednarski [1997]

Theses results showed the ion-selectivity of NF membranes. Bowen & Welfoot [2002] explain this phenomenon by a combination of various effects such as: membrane properties (charged surface and its pores diameter) and size and charge of the ions. Thus high rejection of multivalent salts by NF membrane is caused by their electrostatic repulsion and stopping in membrane pores as a result of size effect [Wang *et al.*, 1997]. Thus larger ions, like magnesium and calcium, are fortunately considered to be nutritionally valuable in food products.

The whey powder solubility was used as a tool for the assessment of overall functional properties of whey powders. Insolubility index values commonly range from 0.4 to 4.3 cm$^3$. In this study, a strongly positive correlation was found between ash content ($r$=0.843) and acidity ($r$=0.709) and insolubility index. This demonstrates, as expected, that the increase in both ash content and acidity of AWP significantly decreased the solubility of AWP samples. These results proved the necessity of demineralisation and deacidification of acid whey before drying. The higher protein content of AWP might be in part accountable for the decrease in whey powder solubility. Protein content was not significantly correlated ($r$=-0.407) with AWP insolubility index. However, this correlation coefficient suggests that protein content is second rate factor in determining the AWP solubility.

The AWP samples from neutralised acid whey were characterised by the lowest solubility, which confirms that ash content is a decisive parameter determining powder solubility. Whey powder manufactured from diafiltered acid whey of non-neutralised NF retentate had the highest solubility as compared to SWP (0.4 cm$^3$).

Low solubility index values of AWP samples are thought to result from extended heat and acid treatments during technological process where denatured proteins and resulting colloidal protein aggregates are of sufficient size to make AWP difficult to dissolve. In addition, lactose in an amorphous form can inhibit protein hydration and reduce powder solubility due to encapsulation and adhering to the active sites of protein [Straatsma *et al.*, 1999; Banavara *et al.*, 2003].

## CONCLUSIONS

The properties of acid whey, namely its composition and acidity, caused technological problems during its evaporation and drying. During these processes, the precipitation of its components was observed to proceed. Acid whey was neutralised and nanofiltered in connection with it. However, a disadvantage of the neutralisation process was the increase in ash content of the final product, which impinged on unacceptable salty taste and overall quality of acid whey powder. Nanofiltration of acid whey was used in order to minimise these defects. The results showed that it was possible to produce spray-dried acid whey powders with reduced ash contents and good properties similar to those of sweet whey powder. This has been achieved by acid whey neutralisation after nanofiltration.

Acid whey composition modification by nanofiltration enhanced its properties. Acid whey powders produced by the application of diafiltration were characterised by the most advantageous properties, even better than those of the sweet whey powders, namely the lowest ash content and acidity and the highest solubility. Solubility of acid whey powders was correlated the most with ash content and acidity.

The results of the present study indicated that the laboratory-produced partially-desalted acid whey powder could be utilised to improve the nutritive value of food products.

## ACKNOWLEDGEMENTS

The study was financed by the State Committee for Scientific Research (KBN), grant No. 5P06G01797C/3594.

## REFERENCES

1. Banavara D.S., Anupama D., Rankin S.A., Studies on physicochemical and functional properties of commercial sweet whey powders. J. Dairy Sci., 2003, 86, 3866–3875.
2. Barrantes L.D., Morr C.V., Partial deacidification and demineralization of cottage cheese whey by nanofiltration. J. Food Sci., 1997, 62, 338–341.
3. Bednarski W., Zagospodarowanie produktów ubocznych. 1997, *in*: Mleczarstwo-zagadnienia wybrane (ed. S. Ziajka), Wydawnictwo AR-T, Olsztyn, Poland (in Polish).

4. Boumba V.A., Voutsinas L.P., Philippopoulos C.D., Composition and nutritional value of commercial dried whey products from feta cheese manufacture. Int. J. Dairy Technol., 2001, 54, 141–145.
5. Bowen W.R., Welfoot J.S., Modelling the performance of membrane nanofiltration-critical assessment and model development. Chem. Engin. Sci., 2002, 57, 1121–1137.
6. Bylund G., Fairy Processing Handbook, Tetra Pak Processing System AB, Sweden, 1995.
7. Glover F.A., Ultrafiltration and reverse osmosis for the dairy industry. Techn. Bull, 1985, 5, NIRO, Reading.
8. IDF, International IDF Standard 129A:1988, Dried milk and dried milk products, Determination of insolubility index.
9. IDF, International IDF Standard 81:1981, Dried milk, Determination of titratable acidity (routine method).
10. Kelly J., Kelly P., Desalination of acid casein whey by nanofiltration. Int. Dairy J., 1995, 5, 291–303.
11. Morr C.V., Ha E.Y.W., Off-flavors of whey protein concentrate: A literature review. Int. Dairy J., 1991, 1, 1–11.
12. Morr C.V., Whey protein functionality: current status and the need for improved quality and functionality. 1990, *in*: Proc. of the Dairy Prod. Technical Conf., Chicago, USA, p. 69.
13. Mulvihill D.M., Grufferty M.B., Production of whey-protein-enriched products. 1997, *in*: Food Proteins and Lipids (ed. S. Damodaran). Plenum Press, New York, pp. 77–93.
14. Nguyen M., Reynolds N., Vigneswaran S., By-product recovery from cottage cheese production by nanofiltration. J. Cleaner Prod., 2003, 11, 803–807.
15. Saltmarch M., Labuza T.P., Influence of relative humidity on the physico-chemical state of lactose in spray dried sweet whey powders. J. Dairy Sci., 1980, 45, 1231–1236, 1242.
16. Straatsma J., Van Houwelingen G., Steenbergen A.E., De Jong P., Spray drying of food products: 2. Prediction of insolubility index. J. Food Engin., 1999, 42, 73–77.
17. van der Horst H.C., Timmer J.M.K., Robertson T., Leenders J., Use of nanofiltration for concentration and demineralization in the dairy industry: model for mass transport. J. Membrane Sci., 1995, 104, 205.
18. Wang X.-L., Tsuru T., Nakao S., Kimura S., The electrostatic and steric-hindrance model for the transport of charged solutes through nanofiltration membranes. J. Membrane Sci., 1997, 135, 19–32.
19. Whiteside P., Miner B., Pye Unicam Atomic Absorption Data Book. 1984, Pye Unicam Ltd., Cambridge.

## CHARAKTERYSTYKA PROSZKU SERWATKOWEGO CZĘŚCIOWO ZDEMINERALIZOWANEGO W WYNIKU NANOFILTRACJI

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W przeprowadzonych doświadczeniach badano skład chemiczny oraz rozpuszczalność proszków serwatkowych otrzymanych z serwatki kwasowej poddanej nanofiltracji I neutralizacji. Uzyskane wyniki wykazały, że modyfikacja składu oraz kwasowości serwatki kwasowej ma istotny wpływ na rozpuszczalność uzyskanych z niej proszków serwatkowych. Spadkowi zawartości popiołu oraz kwasowości towarzyszył wzrost rozpuszczalności proszku serwatkowego. Wykazano istotną zależność pomiędzy zawartością popiołu ($r$=0.843) oraz kwasowością proszku serwatkowego ($r$=0,709) a jego indeksem nie-rozpuszczalności. Przeprowadzone badania wykazały ponadto zasadniczy wpływ miejsca zabiegu neutralizacji serwatki kwasowej w procesie technologicznym na właściwości otrzymanych z niej proszków serwatkowych. Bardziej korzystne okazało się umiejscowienie neutralizacji serwatki kwasowej po procesie nanofiltracji. Najlepszymi właściwościami, nawet lepszymi od proszku serwatkowego z serwatki podpuszczkowej, charakteryzowały się proszki wyprodukowane z serwatki kwasowej poddanej procesowi diafiltracji.