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ANALYSIS OF PHYSICOCHEMICAL PROPERTIES AND FUNCTIONAL QUALITY ATTRIBUTES OF SHELF AVAILABLE MILK POWDERS IN SRI LANKA

Viraj Roshan Weerasingha[1], Janak K Vidanarachchi[2]*, Manju Gunawardena[3] and Rohan Weerasooriya[4]

*Corresponding Author: **Janak K Vidanarachchi,** ✉ janakvid@pdn.ac.lk*

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Physicochemical properties of shelf available milk powders commonly available in Sri Lankan market were investigated and compared them with the international quality standards. Solubility was measured using insolubility index method as one of the major functional properties. Microstructures of the milk powder samples were obtained using Scanning Electron Microscopic (SEM) images and compositional differences were evaluated using Fourier Transform Infra-Red (FTIR) spectroscopy. All the samples had distinct fingerprint characteristics with regard to main nutrients including lipid, protein and carbohydrates according to FTIR spectra. Brand A had very clear high absorption intensities at wavenumbers 1024 cm^{-1} and 3277 cm^{-1} indicating the difference of aliphatic amines for C-N stretch bond and primary or secondary amides or amines for N-H stretch bond, respectively. Imported milk powders showed higher solubility than the locally produced brands and SEM images confirmed presence of uniformly agglomerated powder particles in imported milk powder brands while local products appeared with more fine non-agglomerated particles which can be the main reason for inferior solubility of local powders. Hence, it can be revealed that functionality of milk powders differs significantly due to compositional and structural changes suggesting different processing conditions and quality attributes between locally produced and imported milk powder bands.

Keywords: Physico-chemical properties, Powdered milk, Quality standards, Solubility

INTRODUCTION

Milk is one of the most common and nutritious food sources of human diet which is highly susceptible to spoilage due to the action of spoilage bacteria and naturally occurring enzymes (Grimaud *et al*., 2009). Different methods have been developed with the advances of the dairy sector over the years, for the preservation of milk ensuring the safety and maximum utilization (Singh and Ye, 2010). Among others, milk powder production is the most common processing method used in the industry for preservation of milk. Milk powder is produced by partial removal of water from liquid milk to dryness. Major purposes of milk powder production are preservation of liquid milk and obtain a prolonged shelf life with little or no detrimental changes compared to original liquid milk (Walstra, 1999). Milk powder has become increasingly important product with wide range of end use applications where it requires compositional consistency, desirable physical attributes and functional properties.

1 Department of Animal and Food Sciences, Faculty of Agriculture, Rajarata University of Sri Lanka, Puliyankulama, Anuradhapura 50000, Sri Lanka.

2 Department of Animal Science, Faculty of Agriculture, University of Peradeniya, Peradeniya, 20400, Sri Lanka.

3 Sri Lanka Institute of Nanotechnology, Mahenwatte, Pitipana, Homagama 10200, Sri Lanka.

4 National Institute of Fundamental Studies, Hantana Pedesa Rd, Kandy 20000, Sri Lanka.

Physical and chemical properties of milk powders may subject to changes due to several factors and thereby shelf life and commercial value of the milk powder may deteriorate. Hygienic quality of the raw milk is one of the major factors which determine the powder quality while processing operations such as drying and storage conditions also affect the resultant functionalities of the milk powders (Oliveira *et al*., 2000).

Since milk powder solubility is considered as one of the major functional properties, poor reconstitution of milk powders acts as a limiting factor in different food applications (Martin *et al*., 2007). Insolubility may affect processing difficulties and economic losses as well as negatively affect to other functional properties of milk powders such as emulsification, gelation, foaming and whipping properties (Paracha, 2013). According to the previous approaches, the nature of the insoluble material formed during reconstitution occurred mainly due to casein micelles linked together by fibril-like protein structures (Havea, 2006). In order to address this issue, the influence of different variables on powder solubility have been investigated, such as heat treatment of powder, moisture, protein, mineral content and temperature of reconstitution (Schuck *et al*., 1994; McKenna, 2000; and Dybing *et al*., 2007). Manufacturers are willing to produce nutritious and fresh flavored milk powders with improved functional properties in order to meet the increasing customer demand for high quality dairy products. Hence, these powders should disperse rapidly in water in order to be soluble quickly and completely. The aim of this study was to compare the solubility of the commercially available milk powders in Sri Lanka and assess the physicochemical properties, which could influence on milk powder solubility.

MATERIALS AND METHODS

Experiment was performed using most commonly available four imported milk powder brands (Brand A, B, C, D) and two locally produced milk powder brands (Brand E and F) available in Sri Lankan market, using thirty milk powder sachets as five biological replicates from each brand which have same packed date. A control milk powder sample was prepared by spray drying of freshly droved milk sample using a spray dryer (L-8 No. 7084, Ohkawara Kakohki Co. Ltd., Japan) and used as the authenticated control for the comparisons.

Chemical Property Analysis

Total protein, ash content, fat content and moisture content was measured according to AOAC (2005). Total protein was determined by micro-Kjeldahl method, while ash content was measured by ignition at 550 °C in an electric Muffle furnace. Fat content was determined by Soxhlet extraction method with the modification of 12 hour extended extraction period. Oven dry method was used for determine the free moisture content. Free fatty acid content of milk powder samples was determined using extraction titration method (Vidanarachchi *et al*., 2015). Total Ca content of the milk powders was analyzed using atomic absorption spectrophotometer (AA-6200, Shimadzu, Japan) against standard series of Ca solution.

Water activity of powder samples was determined using a water activity meter (Hygrolab 3, Rotonic Instruments, Japan) and pH determination was carried out using a pH meter (Model: 775249 Eutech, Singapore).

Physical Property Analysis

Bulk density and tapped density of milk powders were determined as described by Sulieman *et al*. (2014). The ratio between bulk density and tapped density of milk powder samples was measured as Hausner ratio. Petroleum ether method was used to determine the particle density of milk powder samples (Gustavo *et al*., 2005).

Solubility of milk powders was measured by insolubility index method, which was described by ISO (2005).

Microstructural Analysis

Scanning Electron Microscopy was used to evaluate the microstructure of the milk powder particles. Milk powder samples were set on the conductive double sided adhesive carbon tape and excess particles were removed using dry air projection. The samples were gold coated by sputtering and analyzed using SEM instrument (EVO 15, Zeiss, German) operating at 10 kV under the magnifications of 250×, 500×, 1000×.

FTIR Spectrum Analysis

Milk powder compositional changes were analyzed using an Attenuated Total Reflection Fourier Transform Infra-Red (ATR-FTIR) spectrometer (Nicolet IS 50, Thermo Scientific, USA) with an Attenuated Total Reflection (ATR) cell (Universal ATR). Around 2-3 mg of the sample was placed on the ATR crystal and absorbance intensity was measured at 4 cm^{-1} resolution in the wave number ranges from 400 to 4000 cm^{-1}. Sample analysis was done at 27 °C and duplication was carried out for each sample.

Statistical Analysis

Significance of each physical and chemical property among milk powder brands were analyzed by one way ANOVA using Complete Randomized Design (CRD). Mean separation was done by Duncan's Multiple Range Test and relationship between each physicochemical property with solubility was analyzed using regression and correlation. Data analysis was done by SAS (9.2 version) software.

RESULTS AND DISCUSSION

Solubility Analysis

Solubility is one of the major functional properties of milk powders, which mainly depend on the chemical composition and physical state of the milk powders. Current study revealed that insolubility index of milk powders ranged between 0.45-0.85 mL (Figure 1) and all the tested milk powder brands had accepted insolubility index values as recommended by CODEX Alimentarius (≤1.0 mL) (CODEX, 2010). The locally produced milk powders had higher insolubility index values while imported milk powders had lower insolubility index values indicating that the imported milk powders possess significantly higher solubility than the locally produced milk powders.

Chemical Property Analysis

Moisture content is rather an important parameter affecting the solubility of the milk powder especially during the storage process, since insolubility of proteins in milk powder could influence by the presence of moisture. Moisture content of milk powder should not exceed 5% and none of the milk powder brands had exceeded the acceptable limits (CODEX, 2010). Imported milk powder brands had significantly higher moisture content than locally produced milk powders except the brand C. Physicochemical stability varies with moisture content during storage and distribution of milk powder, while functional properties like wettability or solubility can also be affected by moisture content (Reh *et al*., 2004). Water activity in milk powder could vary from 0.2 to 0.6 and the results from the current study revealed that water activity of tested milk powder brands ranged between 0.33-0.39. As shown in Table 1, the fat content of milk powder samples varied between 23-29% (w/w). The minimum fat content in whole milk powder should be 26% (w/w) according to CODEX standards (CODEX, 2010). Present study revealed that imported milk powder brand C and locally produced milk powder brand F did not contain the minimum acceptable level of fat content. Le *et al*. (2011)

Figure 1: Insolubility Index Variation of Commercially Available Milk Powders in Sri Lanka

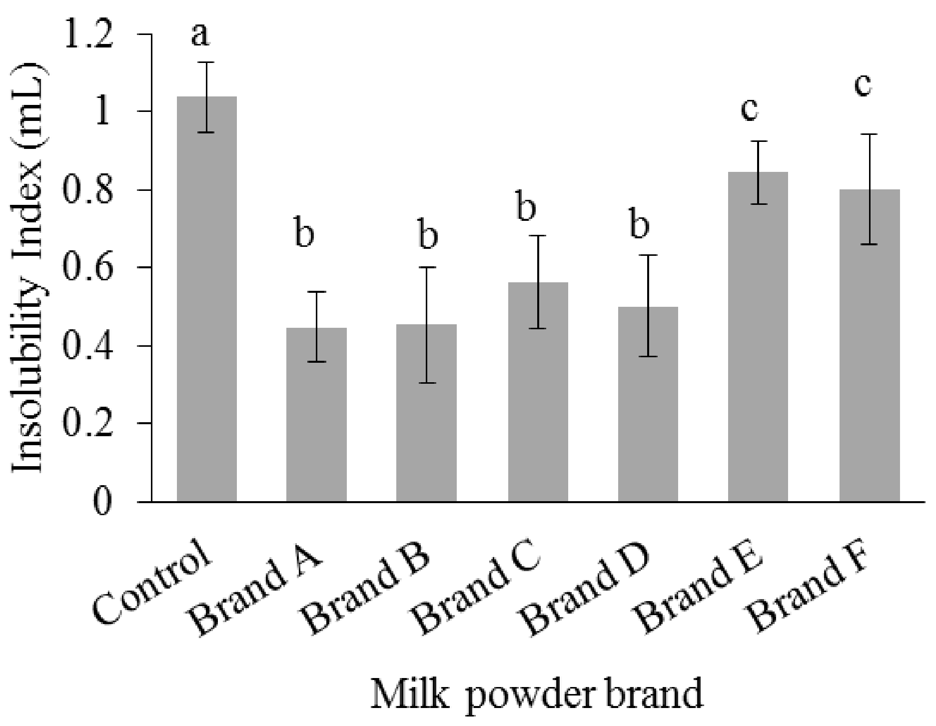


Table 1: Chemical Properties of Whole Milk Powder Available in Sri Lanka (All Values are Presented as the Means ± S.D for Five Replicates Analysis)

Milk Powder Brand	Protein Content (%)	Fat Content (%)	Moisture Content (%)	Water Activity	Ca Content ($mgkg^{-1}$)	Ash Content (%)	pH
Control	29.4 ± 2.5^{a}	17.1 ± 0.4^{d}	1.02 ± 0.2^{c}	0.30 ± 0.01^{d}	7363 ± 135^{a}	6.70 ± 0.4^{a}	7.06 ± 0.13^{a}
Brand A	23.6 ± 0.4^{d}	29.3 ± 1.9^{a}	2.54 ± 0.6^{ab}	0.38 ± 0.02^{a}	4246 ± 470^{c}	5.60 ± 0.6^{c}	6.88 ± 0.09^{ab}
Brand B	23.7 ± 0.5^{d}	26.6 ± 1.0^{ab}	3.10 ± 1.0^{a}	0.39 ± 0.00^{a}	4713 ± 310^{bc}	5.25 ± 0.2^{c}	6.74 ± 0.27^{bc}
Brand C	20.3 ± 0.9^{e}	23.3 ± 1.3^{c}	2.00 ± 1.1^{abc}	0.39 ± 0.02^{a}	4457 ± 622^{bc}	4.05 ± 0.3^{d}	6.74 ± 0.28^{bc}
Brand D	24.3 ± 0.3^{cd}	26.6 ± 1.8^{b}	2.70 ± 1.0^{ab}	0.38 ± 0.02^{ab}	477.1 ± 403^{bc}	5.55 ± 0.3^{c}	6.56 ± 0.24^{c}
Brand E	26.4 ± 1.6^{b}	26.3 ± 1.6^{b}	1.73 ± 0.9^{bc}	0.33 ± 0.01^{c}	567.8 ± 957^{b}	6.28 ± 0.5^{b}	6.91 ± 0.18^{ab}
Brand F	25.6 ± 1.0^{bc}	23.6 ± 1.2^{c}	2.50 ± 0.8^{ab}	0.36 ± 0.01^{bc}	497.3 ± 901^{bc}	5.64 ± 0.2^{c}	7.04 ± 0.17^{a}

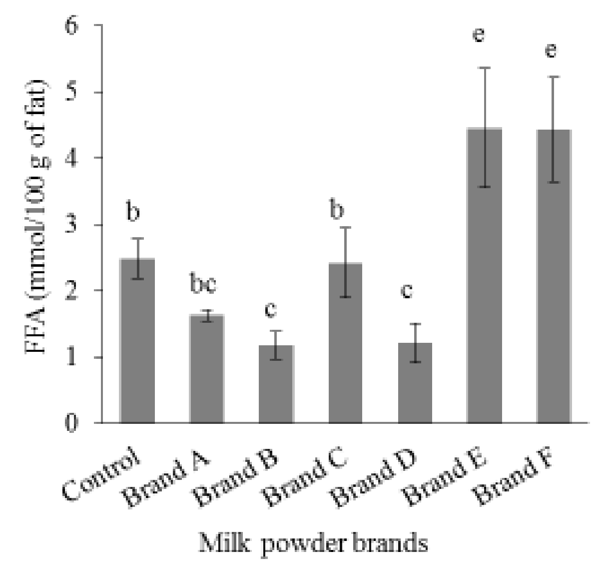
Note: Column mean value with different superscript letters are significantly different at ($P<0.05$).

suggested that high fat content leads to lower solubility in milk powder. However, the present results did not agree with their findings, because milk powder brands with low fat content had shown inferior solubility. It was reported that low water activity, moisture content, fat content and higher insolubility may result due to high spray drying temperatures (Sulieman *et al*., 2014). Since locally produced milk powder possess same characteristics, it could be argued that, higher spray drying temperatures might have used during local milk powder manufacturing process, when compared to imported milk powder manufacturing process.

The acceptable level of protein content in WMP is ranged between 24.5-27% (ADPI, 2002). However, none of the imported milk powder brands have fulfilled the minimum required protein level while all the local milk powder brands were in line with the standard levels (Table 1). Higher casein content together with protein cross linking leads to lower solubility of milk powders. Therefore removal of protein to a certain level could practice by the manufacturers as a solubility improving strategy (Anema *et al*., 2006).

Due to the increased level of free fatty acids, milk powder may prone to higher fat oxidation; resulting poor rewetting properties (Kelly *et al*., 2002). Imported milk powder brands had lower amounts of FFAs levels and those values were within the acceptable limits (1.6-2.3 mmol/100 g of fat) whereas, locally produced brands had two folds higher ($P<0.05$) FFAs levels than that of imported milk powder brands; values were exceeded the standard levels (Figure 2). Previous studies have shown that higher outlet air temperature of spray dryer and degree of lactose crystallinity lead to high free fat levels in milk powder, whereas homogenization assists in reducing free fat levels (Fitzpatrick *et al*., 2004; and Haylock and Dodds, 2009). It has proved that fat oxidation could be accelerated due to low moisture content and current results also agree with same findings (Labuza and Dugan Jr, 1971; and van Mil and Jans, 1991). Quality of the milk and milk products may deteriorate due to the higher FFAs concentrations. Higher somatic cell counts, especially the psychrotrophic and thermo-resistant microflora cause to increase the levels of FFAs due to

Figure 2: Variations of Free Fatty Acids Levels (mmol/100 g of Fat) in Commercially Available Milk Powder Brands in Sri Lanka



increased lipolysis intensity in raw milk and thereby leads to inferior quality and safety of the dairy products (Hanuš *et al*., 2008).

Addition of salt and ion balancing, including pH has major effect on powder solubility and protein stability. Solubility may increase by adding at least one monovalent salt prior to drying (Carr *et al*., 2002). As shown in Table 1, ash content, Ca content and pH of the locally produced milk powder brands were higher ($P<0.05$) than the imported milk powder brands. These three chemical properties are directly linked to each other. High ash content is associated partly with casein protein and high amount of mineral content including calcium and phosphorus like minerals. Babella (1989) found that mineral content of the milk powder has a significant impact on the solubility properties. Milk powder brand A showed the highest solubility and brand E showed the lowest solubility (Figure 1) suggesting that the Ca content of milk powder has negative impact on the solubility properties. It has proved that acidification to lower pH followed by ultrafiltration or addition of calcium chelating agent is carried out to improve the solubility of Milk Powder Concentrates (MPC) (Bhaskar *et al*., 2007). These findings are confirmed by the current study.

Physical Property Analysis

Bulk density is a complex property of instant milk powders which is economically and functionally important. Except the milk powder brand A, bulk density of imported milk powder brands are greater than locally produced milk powders. Bulk density is interrelated with flowability, while particle density is related with sinkability and wettability whereas both these properties affect the rehydration of milk powders. It was reported that milk powders having less than 0.4 g/mL bulk density could leads to high solubility (Harper *et al*., 1963). In the current study, none of the sample had a bulk density less than 0.4 g/mL (Table 2).

Hausner ratio of the milk powder samples which was obtained by dividing bulk density from tapped density of the milk powder samples varied from 1.1 to 1.24 except brand A and E (Table 2). Previous studies have shown that Hausner ratio of 1 to 1.25 indicates better free flowing ability of powder particles, whereas 1.25 to 1.4 indicates fair free flowing ability and ratios greater than 1.4 are cohesive and do not flow well (Sulieman *et al*., 2014). Based on these findings, milk powder samples A and E showed fair free flowing ability while all other brands indicated better free flowing ability. Higher cohesion and attrition force formation among the particles leads to flow resistant due to the presence of high contact surface area among particles (Fitzpatrick *et al*., 2004).

Results from the current study revealed that correlation between solubility and viscosity was not significant ($P>0.05$) and there was no significant difference ($P>0.05$) in viscosity among the tested milk powder brands.

Microstructure of Milk Powder Samples

Examination of microstructure of milk powders provides an insight into why solubility like functional properties differs among different milk powder brands. Scanning electron microscopy can be used to identify the functional properties of milk powders, which are influenced by powder particle morphology. Particle size is an important property, which

Table 2: Physical Properties of Whole Milk Powder Available in Sri Lanka (All Values are Presented as the Means ± S.D for Five Replicates Analysis)

Powder Brand	Bulk Density (g/mL)	Tapped Density (g/mL)	Hausner Ratio	Particle Density (g/mL)
Control	0.32 ± 0.01^{e}	0.36 ± 0.01^{d}	1.12 ± 0.08^{bc}	0.91 ± 0.02^{b}
Brand A	0.43 ± 0.00^{c}	0.55 ± 0.01^{c}	1.30 ± 0.01^{a}	1.15 ± 0.01^{a}
Brand B	0.50 ± 0.02^{ab}	0.61 ± 0.02^{a}	1.21 ± 0.08^{abc}	1.14 ± 0.07^{a}
Brand C	0.55 ± 0.06^{a}	0.59 ± 0.01^{ab}	1.10 ± 0.12^{c}	1.15 ± 0.05^{a}
Brand D	0.50 ± 0.02^{ab}	0.60 ± 0.02^{ab}	1.20 ± 0.01^{abc}	1.20 ± 0.07^{a}
Brand E	0.44 ± 0.00^{cd}	0.58 ± 0.01^{b}	1.32 ± 0.00^{a}	0.99 ± 0.04^{b}
Brand F	0.48 ± 0.01^{cd}	0.60 ± 0.01^{ab}	1.24 ± 0.02^{ab}	1.11 ± 0.03^{a}

Note: Column mean values with different superscript letters are significantly different ($P<0.05$).

can relate to reconstitution, appearance and flow characteristics of milk powders (Hatami and Hejazian, 2011). The amount of the air incorporates between powder particles increase due to agglomeration while compactness and degree of the agglomeration could affect the amount of surface area which contact with water (Fang *et al*., 2012). Hence, it could beneficially affect to enhance the solubility of milk powders by increasing hydrophilic bonds between particles and water molecules. Control milk powder sample didn't show any agglomeration pattern and particles were highly wrinkled when compared to other powder particle surfaces. Powder particles were highly agglomerated and powder surface was much smooth in imported milk powder brands (Figure 3) compared to control sample and other locally produced milk powder brands. Low drying temperature leads to powder particles become more uniform in size and shape with smooth particle surface, while higher drying temperature cause for wrinkled appearance and different size powder particles (Fang *et al*., 2012). As shown in Figure 3, locally produced milk powder particles were not uniform in size, varied the size within a wide range, as well as shape uniformity also relatively lower compared to imported milk powder brands. These compositional characteristics could be due to different manufacturing conditions such as elevated temperature levels during spray drying.

FTIR Spectrum Analysis

Fourier Transform Infra-Red spectroscopy is one of the fastest and direct methods that can be used to evaluate the compositional changes of powdered milk. According to the Figure 4, all the milk powder brands tested in the current study had unique absorption spectral patterns with different absorption intensities.

Absorption spectrum of brand A is different from other available milk powder brands. Milk powder brand A had very clear high absorption intensities with regard to wavenumbers 1024 cm^{-1} and 3277 cm^{-1}. Corresponding functional group and chemical bonds were identified using library spectral data for two different wavenumbers where the absorption intensity is much higher. The chemical bonds corresponding to wave number 1024 cm^{-1} and 3277 cm^{-1} are C-N stretch bond and N-H stretch bond, respectively. The functional groups relate to the C-N stretch bond are aliphatic amine and for N-H stretch bond, it can be primary or secondary amine or amides (Haque *et al*., 2011).

Figure 3: Scanning Electron Microscopy Images of Commercially Available Milk Powders in Sri Lanka Under the Magnification of ×1000 (A, B, C, D are Imported Milk Powders and E, F are Locally Produced Milk Powder Brands)

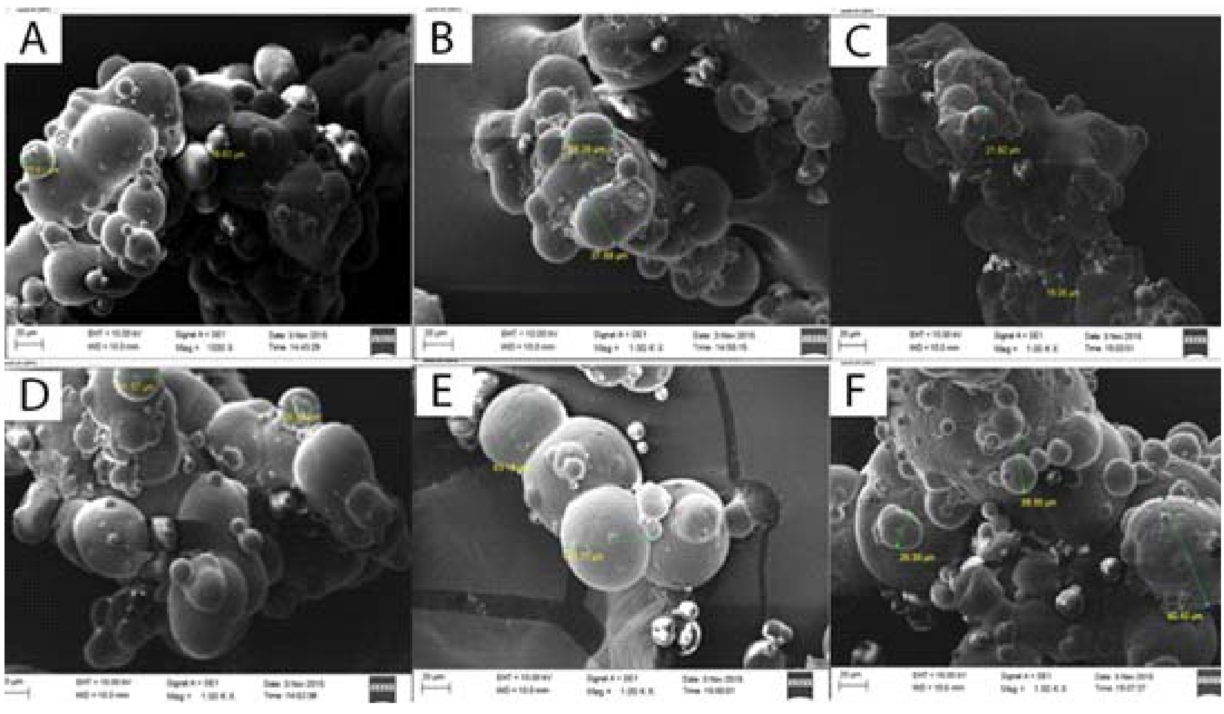
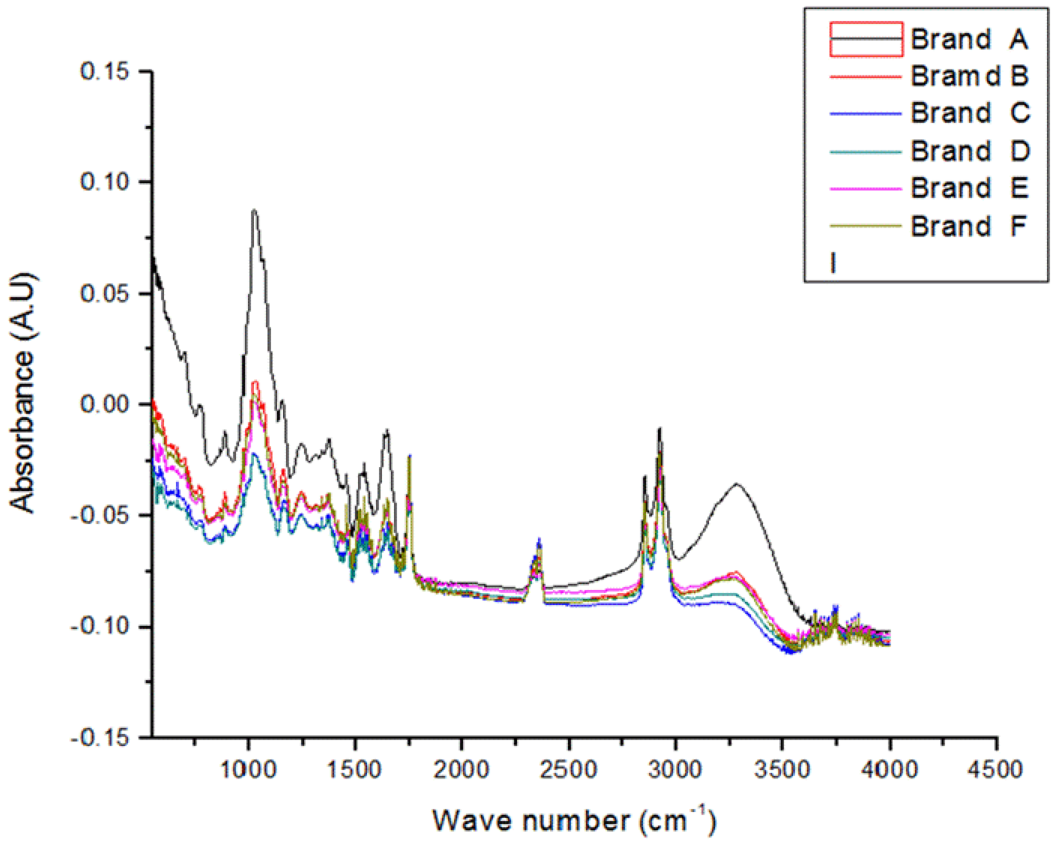


Figure 4: Comparison of Absorption Spectrum, in Wavenumber 400-4000 cm^{-1} of Shelf Available Milk Powder Brands in Sri Lanka



CONCLUSION

Physical and chemical properties of imported and locally produced milk powders tested in the current study are within the internationally accepted specifications except for the free fatty acid content. The current study demonstrated that lower particle density, lower water activity and higher Ca content have association with low solubility properties of milk powders. According to the FTIR analyses, available functional groups of milk powder brand A could have impact on its higher solubility. Scanning Electron Microscopic images confirmed that presence of uniformly agglomerated powder particles in imported milk powder brands while local milk powders appeared with more fine non-agglomerated particles could be one of the main reasons for inferior solubility of locally manufactured milk powders. Hence, it can be revealed that functionality of milk powders differs significantly due to their compositional and structural changes suggesting different processing conditions and quality attributes.

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