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COMPARATIVE ANALYSIS OF FREEZE DRIED AND SPRAY DRIED BEET-ROOT POWDER ACCORDING TO PHYSICO-CHEMICAL, FUNCTIONAL AND COLOR PROPERTIES

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Abstract: *The aim of the research endeavour was to evaluate the powder properties of freeze dried and spray dried beet-root powders.The physical, proximate, color and functional properties such as water activity, wettability, solubility and hygroscopicity were studied. From the comparative analysis between freeze and spray dried beet root powder, better results were found for freeze-dried samples in terms of color, wettability and hygroscopicity. Freeze-dried samples required a longer amount of time to obtain into powder form as compared to spray dried samples. Whereas the solubility and water activity values were found slightly higher in spray dried samples as compared to freeze dried samples. The proximate analysis, such as crude fiber, crude fat, ash and carbohydrate values recorded better result for freeze dried samples as compared to spray dried samples. It can be concluded from this study that the beet root, when dried in the freeze drying process, has better results as compared to the spray drying process.*

Key words: *Beetroot, Spray Drying, Freeze Drying, Hygroscopicity.*

1. Introduction

Dried fruit and vegetable juices have entered the market as a new form of product. Vegetable and fruit powders are being used as an important ingredient in sauces, snacks, extruded cereal products, cake fillings, ice creams and baby food because of their nutritional values [1]. Consumers have found interest in consuming natural products without the presence of any

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artificial additives which has led to various companies shifting to natural pigments from plant or animal origin.

Beetroot, also known as Beta vulgaris, is a root vegetable which contains a great source of iron, vitamin C, manganese, potassium, fiber and folate [9]. Various valuable compounds in beet root are found to have anticarcinogenic properties, including the nitrogenous pigment called betalains [13]. Betalains are most abundantly used in various products to give color along with benefits as they have anti-tumor, antioxidant and anti-diabetic effects. Red violet betacyanin and yellow, orange betaxanthin are the main components of betalains [7]. Betalains being natural pigments are sensitive to external conditions like temperature, oxygen and pH. Beetroot juice is converted into beet root powder for better handling and prolonged shelf life. Despite its medicinal properties and detoxifying nature that improves blood quality and digestion, a few people incorporate beet root powder in their daily diet [19].

Spray drying and freeze drying are the most widely used microencapsulation techniques for converting the product into powder, they preserve the components and protect the core material from the environment by creating a barrier [25]. Spray drying is used to obtain dry powder from a liquid or slurry using hot gas to rapidly dry the product and is preferred for thermally sensitive materials such as food. As higher temperatures are involved in spray drying, there are more chances of quality deterioration of the final product and it also affects the organoleptic properties [23]. Spray drying produces a better quality powder with low water activity in comparison to freeze drying.

The freeze dryer is also called lyophilizer, it is a technique which is based on the sublimation process. This drying is usually regarded as the pleasant process for the production of excellent quality dried powder [22]. But the freeze drying process leads to high production costs, excessive power consumptions and the output is also low. Beetroot juice on spray drying produces a very hygroscopic, low quality and unstable product which makes it undesirable for application in the food industry [21]. Therefore the present investigation was carried out for the comparative analysis of beet root powder obtained through spray drying and freeze drying processes and for the analysis of the physico-chemical and functional properties.

2. Material and Methods

2.1. Sample Preparation

Beetroot samples were procured from a local market. Beetroot was thoroughly washed in running water before use. A screw press juice extractor (M/s Bajaj Process Pack) was employed to obtain the juice from the beetroot and the residual wet pulp was again passed through the extractor to maximize the yield from the sample. Extraction was followed by the filtration of the juice by means of the British Standard Sieve BSS (100 mesh). Pre sterilized glass bottles were used to store the filtered beet root juice. The lab scale spray drying system was used with sample stock with an appropriate dilution factor as the atomizer gets easily clogged because of thicker beet root juice stock [23].

2.2.1. Spray Drying

The solution was passed through the Lab scale spray dryer chamber (500 mm x 215 mm) through a centrifugal pump. The speed of rotation of the pump controls the feed flow rate, which passes from the atomizer nozzle with an inner diameter of 0.5 mm. The inner temperature and feed rate were maintained at two levels 160°C and 400 ml/h as sample (S1) and 180°C and 600 ml/h (S2) respectively. After the spray drying operation, the powder obtained was collected in an insulated glass jar attached at the end of a cyclone and packed in polyethylene pouches. The collected sample was stored till further analysis in a desiccator containing silica gel at 25°C [12].

2.2.2. Freeze Drying

A freeze drier was used at -45°C (F1) and -55°C (F2) for the drying of fresh beetroot. Initially the freshly extracted juice was placed in a uniform container and it was refrigerated for 12 hr at -40 degree Celsius. Then the freeze drying of beetroot juice was carried out using a freeze dryer (M/s Refvac, Vadodara) at a 500 m bar pressure.

2.3. Moisture Loss and Proximate Analysis

Samples were weighed on an hourly basis using electronic weighing balance to measure the weight loss and determining the moisture loss during drying [25]. The proximate analysis of beet root powder such as moisture content, ash, total protein, crude fat, carbohydrates and crude fiber was determined by the official method of AOAC [2].

2.4. Physical Properties of Dried Powder

2.4.1. Bulk Density

It was studied using the process suggested by Balasubramanian et al. [3].

2.4.2. Tap Density

It was studied by filling a measuring cylinder of 100 ml with sample, making sure that a level surface of the sample is formed and then tapped until no further decrease in volume was recorded. The surface was levelled and then weighed. Tap density can be found as product of mass to volume of powdered content [11].

2.4.3. Carr Compressibility Index and the Hausner Ratio

It deals with the compressibility of the food powder, it indirectly affects particle size created after grinding [10]. The Hausner Ratio is obtained by dividing tapping density to bulk density of the sample [11].

2.4.4. Angle of Repose

It is most commonly used to measure the flow properties of samples. It is the resistance of the movement in between the particles and it was studied by using the method devised by Mohite et al. [16].

2.4.5. Coefficient of Friction

It was measured as per process according to the study of Mohite and Sharma [18]. The friction surfaces of wood, aluminium and stainless steel were used in the study. It is assured that the product of weight added in balance to the force required by the sample to just slide on the surface.

2.5. Color Analysis

The Hunter Lab Colorimeter (Hunter Lab, Reston, USA) was used to analyze the color of beetroot powder, which gave L*, a* and b* values. Chroma values were determined to indicate the point of departure of the color from a grey colour of the same lightness [15]. Darkness in color analysis is indicated by the Browning Index (BI) [6]. The L, a and b values were used to estimate the chroma and hue values; they were also used in determining the change in color after the spray and freeze drying processes [5].

$$Chroma = \sqrt{\left(a^2 + b^2\right)} \quad (1)$$

$$Hue\,angle = \tan^{-1}\left(\frac{b}{a}\right) \quad (2)$$

2.6. Functional Properties
2.6.1. Water Activity

Water activity (Aw) is an essential factor to estimate the quality and safety attributes of various food powders. The determination of water activity helps in predicting the presence of possible spoilage microorganisms. Along with measuring microbial spoilage, water activity also affects the activity of enzymes and vitamins in beetroot powder. The water activity of beetroot powder was measured at a temperature of 25°C using an electronic water activity meter [21].

2.6.2. Solubility

The solution of beetroot powder was prepared by dissolving one gram of beetroot powder in 100 ml distilled water and it was then blended using a hand blender. Afterwards it was transferred to a 50 ml centrifuge tube for a centrifugation process of 5 mins at 3000 rpm. After the sample had settled for 30 mins then 25 ml of supernatant was transferred to a pre weighed petri dish. Then it was kept in the oven for 5 hours at 105^{o}C for complete drying. The difference in weight was used to calculate solubility [25].

2.6.3. Wettability

The method used to evaluate wettability was described by Vissottoa et al. [26]. The final value was determined by depositing 1 gram of beetroot powder on the surface of a liquid and calculating the time required by the powder to submerge in the 400 ml of distilled water kept at 25°C.

2.6.4. Hygroscopicity

The hygroscopicity of beetroot powders was estimated by means of the method put forward by Cai and Corke [8]. One gram of sample from each powder sample was placed in an aluminum vial, then it was weighed and equilibrated with salt solution NaCl (providing a relative humidity of 75.3%, according to Greenspan in desiccators at 25°C) [14]. Once the condition of equilibrium had occurred, each and every sample was again weighed. Therefore, from the above described process, hygroscopicity was calculated as a gram of moisture /100 g of solids [23].

3. Results and Discussion
3.1. Moisture Losses and Proximate Analysis Studies

It has been observed that moisture loss decreases at the increase in drying time, the removal of moisture being considerably faster at the beginning of the drying operation because of the higher heat transfer rate [17]. In the starting period of drying, the moisture ratio was high and then it decreased gradually with the increase in temperature [18]. The increase in air temperature increases the removal of moisture and finally it resulted in reducing drying time. The moisture content was found to be 30% wet basis (wb) while the moisture content of dried beet-root juice was found to be 8% (wb). Figure 1 represents the values for the proximate analysis of beet root powder for ash content which was 0.95%, 1.11%, 1.19% and 1.17%, for crude fiber it was 1.51%, 1.21%, 1.77% and 1.81%, for crude fat content it was 0.23%, 0.31%, 0.35% 0.28% and for moisture content it amounted to 2.55%, 2.83%, 3.12% whereas for carbohydrates it was 5.26%, 6.15%. 6.71 and 7.15% was the value for F1, F2, S1 and S2 samples respectively.The influence of the moisture content has its effect on the proximate analysis. It was stated that crude fibre showed the best bond of interaction with the moisture content of dried vegetables. Whereas the role of the ash content with regard to the bonding interaction of varying moisture levels in fruits and vegetables is different and mineral salts were found to prevents moisture reduction at the time of drying. The proximate analysis of spray dried beetroot powder shows increased quantity of carbohydrates, ash content and crude fibre content [19].

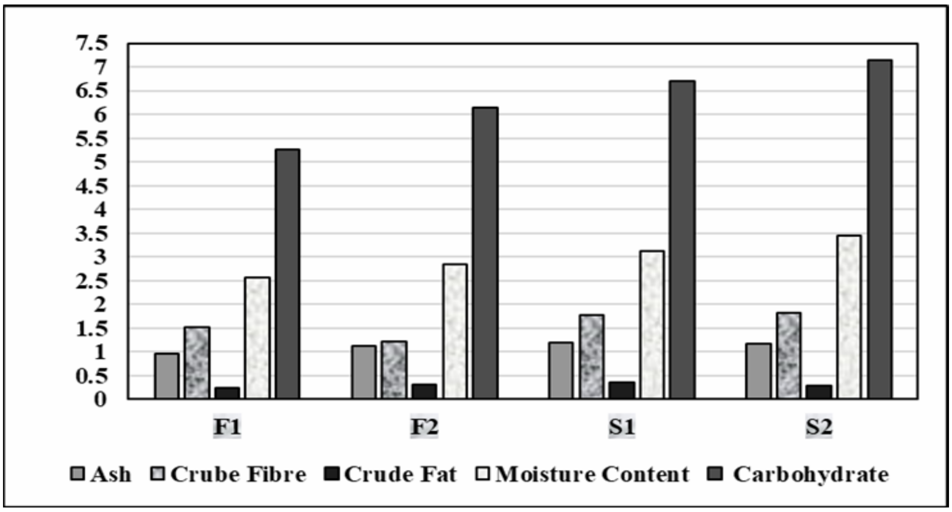

Fig. 1. *Proximate analysis of freeze and spray dried samples: F1- Freeze dried sample at -45°C, F2- Freeze dried sample at -55°C, S1 Spray dried sample at the temperature and feed rate of 160°C and 400 ml/h and S2-Spray dried sample at the temperature and feed rate 180°C and 600 ml/h*

3.2. Physical Property Analysis of Dried Powders

Table 1 represents the physical properties of dried powders. Bulk densities of beet-root powder was found to be 281 kg/m^3, 302 kg/m^3, 309 kg/m^3 and 325 kg/m^3 and tap density was found to be 307 kg/m^3, 326 kg/m^3 352 kg/m^3 and 389 kg/m^3 for F1, F2, S1 and S2 samples, respectively. Bulk density was found to be increasing in all the drying methods.This shows that volumetric increment of particles of dried beetroot powder was responsible for the increasing density value.Similar results were found for turmeric powder by Barnwal et al. [4]. All the factors of the drying process including the temperature, duration and the drying method influenced the bulk density.

Carr's compressibility index (CI) was found to be 13.27, 12.59, 12.33, 12.11 for F1, F2, S1 and S2 samples respectively. Whereas the Hausner ratio was found to be 1.26 (F1), 1.23 (F2), 1.25 (S1) and 1.22 (S2) samples, respectively for freeze dried and spray dried beet root powder samples. Samples which displayed better flowability gave a clear indication that they have a decrease in CI values and Hausner Ratio whereas if the Hausner ratio were increased, it would have represented poor flow properties of spray dried powder samples.

The values of the angle of repose were found to have amounted to 26.12, 27.12, 32.61 and 33.83, for F1, F2, S1 and S2 samples respectively [11]. The highest value of the coefficient of static friction was for wooden surfaces, then aluminium and after that for stainless steel for the samples [17]. The values found an increasing trend from 0.24, 0.31, 0.33 for aluminium; 0.32, 0.39, 0.40 for wooden surfaces; and 0.24, 0.23, 0.27 for stainless steel surfaces, in F1, F2, S1 and S2 samples respectively. There was a close trend found by some researchers such as Barnwal et al. [4] for turmeric powder, [16] for tamarind seeds as per the present results.

Table 1

Physical properties of freeze and spray dried samples

Sample no.	Properties	F1	F2	S1	S2
1	Bulk Density (kg/m3)	281 ±0.55	302 ±0.87	309±0.76	325±0.44
2	True Density (kg/m3)	307 ±0.88	326±0.67	352±0.39	389±0.81
3	Carr's Compressibility Index (CI)	13.27 ±0.12	12.59 ±0.21	12.33±0.21	12.11 ±0.15
4	Hauser ratio	1.26 ±.0.07	1.23±0.03	1.25±0.04	1.22±0.02
5	Angle of Repose	26.12 ±0.27	27.12 ±.0.31	32.61±0.49	33.83 ±0.43
6	Coefficient of static friction (Aluminum)	0.24±0.03	0.31±0.03	0.33±0.02	0.34±0.04
7	Wooden surface	0.32±0.01	0.39±0.03	0.40±0.02	0.44±0.07
8	(Stainless Steel)	0.24±0.05	0.23±0.04	0.27±0.04	0.31±0.03

3.3. Color Analysis

Color values such as L*, a*, b*, Chroma, and hue, for the beet root powder were found as follows. The values obtained were (25.08, 27.8, 8.8, 28.37, 17.74) for F1 (27.8, 31.0, 8.3, 32.38, 14.85) for F2 (20.6, 29.8, 8.6, 31.06, 16.09) for S1 and (21.6, 28.9, 8.04, 29.9, 15.51) for S2 samples respectively. Furthermore, from the analysis it was observed that hue, chroma values were influenced by the temperature of drying of beet root samples. The variation in drying temperature had its influence on the surface colour of samples (Figure 2). The redness value of the spray dried beet-root powder was lower than that of the freeze dried powder due to higher drying temperature [24]. Spray dried beetroot powder had the highest hue angle value but lowest chroma value with a dull color. The freeze drying process led to a very slight color change of the sample which gave a clear indication of the good quality of the samples.

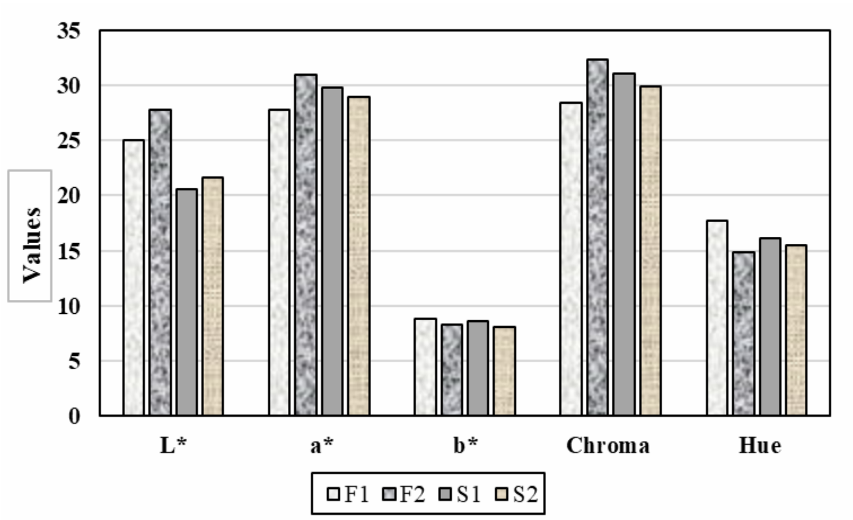

Fig. 2. *Color analysis of freeze and spray dried samples: F1- Freeze dried sample at -45°C, F2- Freeze dried sample at -55°C, S1 Spray dried sample at the temperature and feed rate of 160 °C and 400 ml/h and S2-Spray dried sample at the temperature and feed rate of 180°C and 600 ml/h*

3.4. Functional Properties

The spray dried beetroot powder sample was evaluated for water activity using an electronic water activity meter. It was observed that at a 18.62 temperature, the water activity of beetroot powder was 0.4014 aw. The water activity observed at 17.16 degrees Celsius was 0.9641 aw. The high moisture adsorption might also have promoted the betalains hydrolysis, leading to the formation of betalamic acid, which has a distinct yellow color thus pointing to the fact that hydrolysis was once the primary degradation mechanism of encapsulated betalains, to the formation of betalamic acid in the course of storage at high relative humidities [20]. The degree brix value amounted to 16.7 degree brix [25].

Solubility helps in determining the behaviour of powders in aqueous solutions by estimating the product quality. Powders obtained from freeze drying displayed the highest solubility, the heat induced de-watering of beetroots resulted in a decrease in the solubility of beetroot powders [25]. No significant differences were observed between the solubility of powders obtained from freeze and spray drying processes.

The dissolution of powder components is directly linked to the wettability of the product. It is characterized as the rehydration ability of a powder in water. It was found that the powder took 136 to 281 seconds to become completely vet. The beetroot powder sample was tested for its wettability and it was recorded that the 1g of sample took 4.8, 5.5, 5.9 and 6.2 seconds to submerge in 400ml of distilled water. The absorption of water is a vital aspect for powder reconstitution since it can result in caking and thus reducing dispersibility. The values obtained for hygroscopicity varied from 20.15 to 23.18%, and they were influenced considerably by the elements present in the beetroot powder samples.

4. Conclusion

The comparative study of beet root powder dried by freeze drying and spray drying processes was investigated on the basis of different powder properties.

1. Freeze dried and spray dried beetroot powder showed slight differences in the proximate analysis, including ash content, crude fibre, crude fat and carbohydrates;
2. It was found that physical properties like bulk density, tap density, coefficient of friction and angle of repose showed significant differences in freeze dried and spray dried beet root powder. Freeze dried beet powder had higher values as compared to spray dried powders;
3. Beet root powder flow properties were found to be influenced by drying techniques, spray dried beet root powder was less hygroscopic as compared to freeze dried beet root powder;
4. Color properties of freeze dried beet root powder showed better results when compared to spray dried beet root powder;
5. From the study it can be concluded that even though the freeze drying process took more time with regard to the drying process, the final quality of the beet root powder was the best.

References

1. Aggrawal S., Mohite A.M., Sharma N., 2018. The maturity and ripeness phenomenon with regard to the physiology of fruits and vegetables: A review. In: Bulletin of the Transilvania University of Brasov, Series II: Forestry, Wood Industry, Agricultural Food Engineering, vol.11(60), no. 1, pp. 77-88.
2. AOAC, 2000. Official methods of analysis. In: Association of Official Analytical Chemists. Inc., 17th Edition, Arlington, Virginia, U.S.A..
3. Balasubramanian S., Singh K.K., Mohite A.M. et al., 2012. Physical properties of cinnamon bark. In: Journal of Spices and Aromatic Crops, vol. 21(2), pp 161-163.
4. Barnwal P., Mohite A.M., Singh K.K. et al., 2014a. Selected physico-mechanical characteristics of cryogenic and ambient ground turmeric. In: International Agrophysics, vol.28, pp. 111-117.
5. Barnwal P., Mohite A.M., Singh K.K. et al., 2014b. Effect of cryogenic and ambient grinding on grinding characteristics of cinnamon and turmeric. In: International Journal of Seed Spices, vol. 4(2), pp. 26-31.

6. Barnwal P., Singh K.K., Mohite A.M. et al., 2015. Influence of cryogenic and ambient grinding on grinding characteristics of fenugreek powder: a comparative study. In: Journal of Food Processing and Preservation, vol. 39(6), pp. 1243-1250.
7. Bazaria B., Kumar P., 2016. Optimization of spray drying parameters for beetroot juice powder using response surface methodology (RSM). In: Journal of the Saudi Society of Agricultural Sciences, vol. 17(4), 9p.
8. Cai Y.Z., Corke H., 2000. Production and properties of spray-dried *Amaranthus betacyanin* pigments. In: Journal of Food Science, vol. 65(7), pp. 1248-1252.
9. de Carmo E.L.D., Ribeiro Teodoro R.A., Campelo Félixb P.H.C. et al., 2017. Stability of spray-dried beetroot extract using oligosaccharides and whey proteins. In: Food Chemistry, vol. 249, pp. 51-59.
10. Hausner H.H., 1967. Friction conditions in a mass of metal powder. In: International Journal of Powder Metallurgy, vol. 3(4), pp. 7-13.
11. Jamil Z., Mohite A.M., Mishra A. et al., 2020. Selected engineering properties and drying behavior of tendu (*Diospyros melanoxylon* Roxb.) fruit. In: Current Researh in Nurition and Food Science, vol. 8(2), pp. 622-629.
12. Jinapong N, Suphantharika M, Jamnong P., 2008. Production of instant soy milk powders by ultrafiltration, spray drying and fluidized bed agglomeration. In: Journal of Food Engineering, vol. 84(2), pp. 194-205.
13. Koul V.K., Jain M.P., Koul S. et al., 2002. Spray drying of beet root juice using different carriers. In: Indian Journal of Chemical Technology, vol. 9(5), pp. 442-445.
14. Kurozawa L.E., Morassi A.G., Vanzo A.A. et al., 2009. Influence of spray drying conditions on physicochemical properties of chicken meat powder. In: Drying Technology, vol. 27(11), pp. 1248-1257.
15. Mohite A.M., Chandel D., 2020. Formulation of edible films from fenugreek mucilage and taro starch. In: SN Applied Sciences, vol. 2(11), article no. 1900.
16. Mohite A.M., Mishra A., Sharma N. 2019. Influence of different grinding processes on powder characteristic of tamarind seeds. In: Agricultural Research, vol. 9(2), pp. 262-269.
17. Mohite A.M., Mishra A., Sharma N., 2016. Equilibrium moisture content and drying behaviour of tamarind seed under thin layer condition. In: International Journal of Seed Spices, vol. 6(2), pp. 18-22.
18. Mohite A.M., Sharma N., 2018. Drying behaviour and engineering properties of lima beans. In: Agricultural Engineering Journal – The CIGR e-journal, vol. 20(3), pp. 180-185.
19. Murlidhar I., Thorat S.S., Kotecha P.M. et al., 2017. Nutritional assessment of beetroot (*Beta vulgaris* L.) powder cookies. In: Asian Journal of Dairy and Food Research, vol. 36(3), pp. 222-228.
20. Ng M.L., Sulaiman R., 2018. Development of beetroot (*Beta vulgaris*) powder using foam mat drying. In: LWT – Food Science and Technology, vol. 88, pp. 80-86.
21. Rafiq S.I., Singh S., Saxena D.C., 2016. Physical physico-chemical and anti-nutritional properties of horse chestnut (*Aesculus indica*) seed. In:

Journal of Food Measurement and Characteristic, vol. 10(2), pp. 302-310.

22. Ratti C., 2001. Hot air and freeze-drying of high-value foods: A review. In: Journal of Food Engineering, vol. 49(4), pp. 311-319.
23. Santhalakshwy S., Bosco S.J.D., Francis S. et al., 2015. Effect of inlet temperature on physicochemical properties of spray-dried jamun fruit juice powder. In: Powder Techology, vol. 271, pp. 37-43.
24. Sara M.S., Awad Amira M. et al., 2019. Usage of red beet (*Beta vulgaris* L.) and roselle (*Hibiscus sabdarriffa* L.) in jelly production. In: International Journal of Eniviroment, vol. 8(2), pp. 142-150.
25. Seth D., Mishra H.N., Deka S.C., 2017. Functional and reconstitution properties of spray-dried sweetened yogurt powder as influenced by processing conditions. In: International Journal of Food Properties, vol. 20(7), pp. 1603-1611.
26. Vissottoa F.Z., Jorgea L.C., Makitaa G.T. et al., 2010. Influence of the process parameters and sugar granulometry on cocoa beverage powder steam agglomeration. In: Journal of Food Enginnering, vol. 97(3), pp. 283-291.