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Influence of moisture content on the flow properties of *basundi* mix

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ABSTRACT

In this study, flow and flow-related properties namely morphology, angle of repose, moisture sorption, glass transition and sticky point temperature of *basundi* mix powder were determined. The bulk and tapped densities decreased from 690.71 to 622.71 kg/m^3 and 819.10 to 729.31 kg/m^3, respectively as moisture content increased from 3 to 9% (d.b.). Cohesiveness, expressed in terms of Hausner ratio and Carr index, increased with increase in moisture content. The sticky point temperature also decreased from about 40 °C at 2% moisture content to 10 °C at 8% moisture content, suggesting that the product was very much susceptible to caking. The basic flow energy values increased from 783.39 kPa at 3% moisture content to 1883.2 kPa at 9% moisture content. Shear tests showed that *basundi* mix was relatively more flowable at 3% (or less moisture content) than at 9% moisture content. It could be concluded that dynamic flow tests were better in characterizing flowability of *basundi* mix than static flow indicators such as angle of repose and Hausner ratio. Overall, *basundi* mix could be classified as a cohesive material, and to have better flowability and less stickiness, this powder is recommended to be stored at <30 °C and 40% RH. This characterization will help to understand the behavior of *basundi* mix during processing, and will be useful in the design of handling, processing and storage equipment.

1. Introduction

Flow of powders is defined as the relative movement of bulk particles among neighbouring particles or along the container wall surface. Dairy powders are largely diverse on their own and most of the dairy based ingredients and ready-to-mix materials are manufactured in powder form for their convenience in production, handling and storage. Flowability of powders is inherently complex as there are many factors that influence the response of powders when moved [1]. Bargale et al. [2] mentioned that bulk powders that were subjected to a series of static and dynamic loads during handling, transport, processing, and storage were inherently affected, and therefore, might undergo change in their physical and flow properties during discharge from hoppers and silos.

Basundi, a heat-desiccated milk sweet, is popular in India and its neighbouring countries. Preparation involves open pan (shallow kettle) concentration of sweetened milk (sugar 6% of milk) till the total solids content of 28–32% is achieved. *Basundi* thus prepared has a relatively thick creamy consistency, white to light caramel colour, sweetish caramel aroma, and soft flaky texture. The conventional method of preparing *basundi* from milk is tedious and cumbersome. As an alternative to preparing this product at household level from milk, low-moisture *basundi* mix powder has become a product of commercial significance. The dry *basundi* mix remains stable at ambient temperature [3] and offers reduced costs in the supply and delivery chain.

It has been proved that bulk properties such as moisture content, density, shape, particle size distribution, composition, temperature, relative humidity (RH), and storage time influence the flow properties of particulate materials [4]. Similarly, Freeman [1] showed that the powder flowability was a consequence of the combined effects of various engineering, chemical and environmental variables.

With high variability within powders due to their differences in physical and chemical characteristics, many traditional powder flow characterization techniques result in poor repeatability and do not simulate dynamic processing conditions. For example, bulk properties of powders are affected by air to some extent as the space between the particles becomes filled with air. Similarly, moisture sorption, glass transition and stickiness are related to flow of powders. The amount of air present influences how the particles interact with each other, which consequently impacts directly upon the flow properties. Some powders can be readily aerated and only require a small amount of air and energy to transform the powder bulk into a fluidised bed, wherein the powder behaves as a fluid. The Freeman's FT4 powder rheometer is a modern and recent tool to analyze the complex flow behaviours of food powders by dynamic testing. Alisa et al. [5] and Leturia et al. [6]

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demonstrated the use of dynamic flow properties of powders for accurate flow characterization.

Understanding the flow behaviour of bulk granular materials as influenced by moisture content is therefore important for ensuring steady and reliable flow [7] during handling and processing operations [8,9]. The flow properties of dry *basundi* mix have not been studied. Therefore, this study was conducted with the objective of determining the flow-specific and dynamic flow properties of *basundi* mix and characterizing it in terms of basic, aeration and shear flow properties.

2. Materials and methods

A nationally known brand of *basundi* mix powder was procured from the local market and used in this study. This product contained skim milk powder (SMP), refined wheat flour, sugar and hydrogenated vegetable oil as basic ingredients. The proximate composition of *basundi* mix was analyzed to determine the major constituents.

2.1. Proximate composition

The moisture content of *basundi* mix was determined by AOAC method 927.05 [10], and all values reported in this work are based on dry basis. Exactly 2 g of sample was used for analysis of moisture content. Fat content was determined by AOAC method 922.09 [10]. The protein content was estimated using Micro-Kjeldahl method (method 930.29 of AOAC) [10]. The total ash content of the sample was determined gravimetrically by AOAC method 930.30 [10], while the carbohydrate content was calculated from the difference to 100. All the chemical analyses were done in triplicate.

2.2. Moisture conditioning

The flow properties of the *basundi* mix were analyzed at moisture contents of 3, 6 and 9% (d.b.). The samples were adjusted to the desired moisture contents either by drying in a hot air oven at 50 °C [11] or by sprinkling calculated amounts of de-ionized water using a handheld sprayer, followed by mixing the powder and allowing the samples to equilibrate for 5 h in airtight containers.

2.3. Morphology

The microstructure of *basundi* mix was examined using a scanning electron microscope (SEM) (model: JSM-6390LA, Japan Electron Optics Laboratory, Tokyo, Japan). The powder was spread as a thin layer on a glass plate and one layer of powder was made to stick to the stubs, and the samples were sputter-coated (model: JFC-1600, Japan Electron Optics Laboratory, Mitaka, Tokyo, Japan) with gold to make them conductive before examination. After coating, the samples were scanned under vacuum to get the microstructural images. Acceleration voltage used was 10–15 kV. Images were acquired at 500×, 1500× and 3000× magnifications and at 1280 × 960 pixels resolution.

2.4. Particle size distribution

Particle size is an important physical property of milk powder, and it can be related to its appearance, reconstitution and flow characteristics. As *basundi* mix agglomerated heavily at higher moisture contents, the particle size distribution was determined by wet method. In this method, the particle size was measured by laser diffraction technique using Malvern Mastersizer (model: Mastersizer Hydro 3000, Malvern Instruments Ltd., Malvern, Worcester, UK). About 10 g of powder was mixed with 100 mL of isopropyl alcohol, and 2–3 mL of above slurry was poured into the sample holding well. Isopropyl alcohol was preferred as the dispersion medium because it provided uniform dispersion of the particulates without sedimentation. The sample was allowed to spread over the analyser attached with lens. The angular deflection was measured through detector sensors placed at different positions. The observed angle of deflection was then converted automatically to particle size in terms of $d_{0.1}$, $d_{0.5}$ and $d_{0.9}$ diameters. At least three repeat measurements were done for each sample, and the mean values of $d_{0.1}$, $d_{0.5}$ and $d_{0.9}$ diameters were calculated. The span, which was a measure of width of the size distribution, was also determined (Eq. (1)).

$$Span = \frac{d_{0.9} - d_{0.1}}{d_{0.5}} \tag{1}$$

2.5. Bulk and tapped densities

The ASTM standard D7481-09 [12] was followed to determine the bulk and tapped densities of *basundi* mix. A 250 mL graduated cylinder was taken, and the tarred weight was noted. A funnel was placed over the cylinder opening, and the powder was allowed to flow freely through the funnel up to 250 mL mark. The cylinder was tapped gently three times. The weight of the cylinder together with powder was recorded. The net weight of 250 mL of powder was calculated, and the bulk density (mass/volume) was expressed as kg/m^3. For tapped density, the powder was filled in the same cylinder and it was tapped 500 times in a tapped density tester (model: Thermonik, Campbell Electronics, Mumbai, India). The volume of sample was then measured, and from the mass of sample, the tapped density was calculated. The measurement was repeated eight times, and the mean values are reported.

2.6. Particle density and porosity

The particle density of *basundi* mix was determined as per the method outlined by Pushpadass et al. [13]. Exactly 5 g of powder was weighed and transferred into a 50 mL graduated centrifuge tube with an air-tight stopper. Subsequently, 25 mL of petroleum ether was added to the tube, and it was shaken well for 1 min until all the powder particles were uniformly dispersed. All powder particles on the wall of the tube were rinsed down with additional 3 mL of petroleum ether (28 mL in total). The tube was then vigorously agitated on a vortex mixer for 5 min. The total volume of petroleum ether along with suspended powder particles was noted. The particle density of *basundi* mix was calculated as follows:

$$\text{Particle density }(\text{kg/m}^3) = \frac{\text{Weight of sample}}{\text{Total volume of petroleum ether with suspeneded powder (mL)} - 28}. \tag{2}$$

Porosity (ε) of *basundi* mix at different moisture contents was calculated using the relationship between tapped (ρ_{tapped}) and particle ($\rho_{particle}$) densities of the powders as follows:

$$\text{Porosity}(\%) = \frac{(\rho_{particle} - \rho_{tapped})}{(\rho_{particle})} \times 100. \tag{3}$$

2.7. Flow indicators

Flow indicators of *basundi* mix was evaluated in terms of Carr index (CI) [14] and Hausner ratio (HR) [15], respectively. Both CI and HR were calculated from the bulk (ρ_{bulk}) and tapped (ρ_{tapped}) densities of the powder as follows:

$$CI(\%) = \frac{(\rho_{tapped} - \rho_{bulk})}{(\rho_{tapped})} \times 100 \tag{4}$$

$$HR = \frac{(\rho_{tapped})}{(\rho_{bulk})}. \tag{5}$$

2.8. Angle of repose

The angle of inclination of the free surface to the horizontal of a bulk solids pile is measured by angle of repose. It is one of the primary parameters of granular materials, which indicates the friction between the particles. Angle of repose is closely related to particle density, size, surface area, shape and coefficient of friction [16]. An optical imaging method was used to measure the static angle of repose. Free-standing piles of particulates were formed by allowing the powders to pass through a fixed funnel of 15 cm top diameter and 2.5 cm bottom opening. The images of *basundi* mix piles were taken using a digital camera (model: fz8, Panasonic Corporation, Osaka, Japan) at moisture contents of 3, 6 and 9%. The 'Drop Snake' tool of ImageJ plugin (ver. 1.45) was used to measure the angle of repose from the images after converting them to grey-scale.

2.9. Moisture sorption behaviour

The static gravimetric technique based on isopiestic transfer of water vapour was followed to obtain the moisture sorption isotherms [17]. About 1 g of each powder, weighed to an accuracy of 0.1 mg, was taken in beakers of 25 mL volume. Each of these beakers was placed inside 50 mL beakers, and they were kept inside desiccators containing the salt slush. The desiccators were placed in a stability chamber (model: SC-10 Plus, REMI Sales and Engineering, Mumbai, India) at temperatures of 10, 25 and 40 °C for 2 days before the samples were kept. The samples in desiccators were equilibrated in the stability chamber for 45 days, and six determinations of moisture content at each water activity (a_w) and temperature were used to construct the moisture sorption isotherms (MSI). The equilibrium moisture content (EMC)-a_w data were fitted to the Guggenheim-Anderson-de Boer (GAB) model (Eq. (6)).

$$M_w = \frac{M_0 C K a_w}{(1-Ka_w)(1-Ka_w+CKa_w)} \tag{6}$$

where M_o was the monolayer moisture content, M_w was the EMC and '*C*' and '*K*' were the adsorption constants which were related to the energies of interaction between the first and further sorbed molecules at the individual sorption sites. It is well-known that EMC and monolayer moisture content influence the shelf-life stability of food powders.

2.10. Glass transition temperature

The temperature which characterizes the stickiness and caking tendencies in food powders are glass transition temperature (T_g) and sticky point temperature (SPT) [18]. The T_g of *basundi* mix was determined using a differential scanning calorimeter (model: DSC822e, Mettler Toledo, Greifensee, Switzerland). Samples weighing 10 ± 0.2 mg were heated under nitrogen atmosphere from 0 to 90 °C, at a heating rate of 3 °C/min. The DSC was calibrated using indium as the standard, and measurements were taken against an empty aluminium pan as the reference. Scans were done in duplicate on each sample. The onset of T_g was calculated using the Star SW 8.10 software supplied by the equipment manufacturer.

2.11. Sticky point temperature

The SPT, at which stickiness or caking occurs in the product, was determined using a Brookfield viscometer (Model: RVDV-II + Pro, Brookfield Engineering Laboratories, Middleboro, MA) attached with a helipath stand and t-bar (T-F) spindle [13]. Before measurement, the moisture contents of *basundi* mix were adjusted to 2, 4, 6, 8 and 10% d.b. Isothermal measurements were carried out from 10 to 80 °C at 10 °C intervals. At all moisture contents, exactly 50 g of sample was weighed on to 100 mL beakers, tapped three times and placed in a water bath for 45 min to equilibrate them to the test temperature. The beaker top was covered with aluminium foil to prevent loss of moisture (except that a hole was made on the foil to place the spindle during testing). The torque required for rotation of the spindle in the powder was measured at a spindle speed of 0.5 rpm. Data were acquired for 240 s at 10 s intervals using Rheocalc software (v. 3.1.1) supplied by the viscometer manufacturer. The average torque requirement at each moisture content-temperature combination was calculated from the data points between 30 and 120 s. The temperature at which the torque showed a drastic increase in value was identified as the SPT at that moisture content [19]. The experiment was repeated three times and the mean values were computed.

2.12. Dynamic flow properties

The FT4 powder rheometer (model: FT4, Freeman Technologies, Tewkesbury, UK) was used to analyze the flow properties of the powders in terms of energy required to make them flow. A detailed description of this instrument and its use in powder characterization are found in Freeman [20] and Leturia et al. [6]. The FT4 instrument employs a helical blade to establish a specific flow pattern in a precise volume of powder and estimate the ability of the sample to resist the motion of the blade. The dynamic flow properties were analyzed under packed bed, free surface and aerated conditions. All tests were carried out with a 48 mm helical blade and 50 mm dia. split vessel made of borosilicate glass. Basic flow energy (BFE), stability index (SI), flow rate index (FRI), specific energy (SE) and compressibility were measured. The structure of FRI and SI tests was simply a combination of four conditioning and test cycles and seven conditioning and test cycles, respectively as shown in Fig. 1 (Instruction manual W7013, Freemans Technology, Tewkesbury, UK).

2.12.1. Basic flow energy

BFE is described as the energy required to establish a specific flow pattern for a precise volume of particulate solid materials. BFE corresponds to the stabilized flow energy needed to displace a conditioned powder sample during downward movement of the probe in testing [6]. The BFE (mJ) of the powders was calculated from the work done in moving the blade through the powder from the top of the test vessel to the bottom at the rate of 5° helix downwards and at a blade tip speed of 100 mm/s. This test measures the effect of moisture and agglomeration on dynamic powder flow.

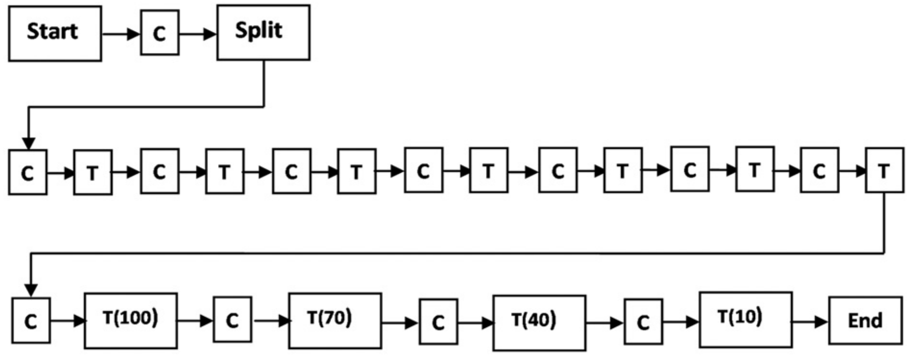
2.12.2. Stability index

Stability index assesses if the powder changes its form such as agglomeration or segregation under identical and repetitive conditioning and test cycles of powder flow. External variables such as flow rate or air velocity do not affect the output of the test. For stability measurement, all test cycles were conducted at 100 mm/s blade tip speed with the blades moving transversely down the vessel. The SI was calculated using the following relationship:

$$\mathrm{SI} = \frac{\text{Energy at test 7}}{\text{Energy at test 1}}. \tag{7}$$

2.12.3. Flow rate index

The rate at which powders are transported and managed in the production or processing facility differs from location to location in the process line. As flow rate depends on properties like cohesion, aeration, particle size, agglomeration, etc., bulk powders are responsive to change in flow rates. To understand the whole process during handling and transportation, characterization of flow rate is particularly important. To estimate the flow rate, the flow energy of *basundi* mix was measured at four different blade tip speeds during downward movement of the blade. The test started with subjecting the powder



where C = conditioning cycle

T = test cycle

(n) = ‘n’ is the blade test speed during the cycle (mm/s)

Split = splitting of vessel to provide precise volume of powder for measurement

Blade tip speed is 100mm/s where not defined in brackets

Fig. 1. Test sequence of flow measurement.

to a standard flow rate of 100 mm/s (Test 1) followed by measuring at 70 (Test 2), 40 (Test 3), and 10 mm/s (Test 4) blade speeds. The FRI was calculated from the flow energy using Eq. (8) [20].

$$\mathrm{FRI} = \left(\frac{\text{Flow energy at test 4}}{\text{Flow energy at test 1}}\right). \tag{8}$$

2.12.4. Specific energy

In contrast, the SE (mJ/g) was derived during the upward traverse of the blade through the powder bed using a gentle shearing and lifting mode of displacement. It provides an indication of the flow properties of the powder in a loosely packed and unconfined state. The value of SE was then determined by dividing the energy by the split mass.

$$\mathrm{SE(mJ/g)} = \frac{(\text{Up energy cycle 6} + \text{Up energy cycle 7})}{2 \times \text{Split mass}} \tag{9}$$

The conditioning process utilized the standard FT4 helical blade, which moved the powder in a gentle way in order to establish a standardized packing state and aeration of powders.

2.12.5. Compressibility

Compressibility is a measure of change in density as a function of applied normal stress [21], which in turn relates the mechanical compaction that happens in powders during handling, storage and transportation. That is, compressibility indicates the percentage by which the bulk density increases when a normal stress is applied to the sample. Though compressibility is not a direct measure of flowability, it can be used to understand the behaviour of powders in various process or handling environments. This bulk property is influenced by particle size distribution, cohesivity, particle stiffness, particle shape, and particle surface texture. The compressibility tests were carried out using venting piston assembly, which compressed the sample under increased normal stresses. The sample was placed in a 50 mL cylindrical vessel and normal stresses progressively increasing from 0.5 to 15 kPa was applied to consolidate the samples. Each normal stress was maintained for 25 s on the target stress to reach equilibrium. The distance travelled by the piston was recorded, and the compressibility percentage was calculated from the percentage change in volume.

2.12.6. Aeration

Powders are porous medium with interparticular spaces filled with air. The amount of air influences the particle to particle interaction, and thus, has a direct impact on flow properties. The ability of a powder to be influenced by air is a very good indicator of powders' absolute cohesivity. The energy required to establish a given flow pattern in the powder is a direct function of how easily the flow of gas separates the individual particles. Thus, easily aerated powders have better flowability as small amount of energy can initiate fluid flow. Using FT4, the aeration properties of *basundi* mix was measured as a change in flow energy with introduction of air through the base of the column of powder. During the test, no air was introduced at first run. The velocity of air passing through the powder bed was gradually increased, and the energy used by the blade during downward motion was recorded. The test was conducted at velocities from 2 to 10 mm/s for *basundi* mix with an increment of 2 mm/s in a single run. The blade tip speed was maintained at 100 mm/s, and the aeration ratio (AR) for individual powders was calculated as

$$\mathrm{AR} = \left(\frac{\text{Flow energy at the air velocity of 0 mm/s}}{\text{Flow energy at the air velocity of 10 mm/s}}\right). \tag{10}$$

2.12.7. Shear properties

The shear properties provide information as to whether *basundi* mix will flow or bridging and blockages occur while passing through the process line. Shear tests were conducted using a 48 mm FT4 shear cell that induced both vertical and rotational stresses. The normal stress was maintained constant at 9 kPa throughout the measurement. A series of yield points for a range of reducing normal stresses was determined and the yield loci were obtained. Different powder properties such as cohesion (kPa), unconfined yield strength (UYS), angle of internal friction (AIF, deg), and flow function (ff_c) (or friction coefficient) were measured from the relationship between normal and shear stress. The UYS was obtained from the stress circle tangential to the yield locus and passing through the origin. The ff_c value was calculated as the ratio of major principal stress (MPS) and UYS.

$$ff_c = \frac{\sigma_1}{\sigma_c} = \frac{MPS}{UYS} \quad (11)$$

where σ_1 and σ_c were MPS and UYS in kPa.

2.12.8. Wall friction

The wall friction test is similar to the shear cell test, but instead of forcing the powder to shear against itself, a disc of a known material and surface roughness is forced to shear against the surface of powder bed whilst being subjected to varying normal stresses. Wall friction properties are important for understanding the flowability of a consolidated powder, which was at rest previously, in relation to the wall material of its container. Knowledge of wall friction is critical because it is an important parameter for determining the minimum hopper angle required for a consistent and reliable flow [22]. Wall friction properties were evaluated using standard FT4 blade with vented piston. The split vessel used for compression test was then used to take a precise volume of powder. Initially, the powders were prepared by conditioning and pre-conditioning using standard FT4 blade and vented piston. After taking the precise volume of powder by splitting, the piston was then replaced with the wall friction head and a relevant surface roughness disc having a surface roughness of 0.28 Ra (surface roughness) was mounted. Wall friction head was attached to the FT4 powder rheometer and the samples were subjected to vertical and rotational stress. As the powder bed resisted the rotation of the wall friction head, the torque increased until the resistance was eventually overcome. The wall friction head was made to rotate at a fixed velocity for a pre-determined period of time, and the torque required to maintain this rotational momentum was measured as shear stress. The relationship between normal stress (σ_w) and shear stress (τ_w) was the wall friction angle (ϕ) [6]. It is the angle subtended by the yield locus, and its magnitude depends on the properties of both the powder and material of construction and its surface finish. A larger wall friction angle (deg) indicated a higher resistance to flow of the specific powder across the test surface. The wall friction angle of the samples was calculated using Eq. (12).

$$\phi = \tan^{-1}\left(\frac{\tau_w}{\sigma_w}\right) \quad (12)$$

where τ_w was the shear stress and σ_w was the normal stress in kPa.

2.13. Statistical analyses

Physical properties such as particle size distribution, densities, angle of repose, etc. were analyzed at least in quadruplicate, and the mean values were computed. The glass transition temperature, sticky point temperature and all powder flow tests were determined in triplicate and new powder samples were used for each test. The physical and flow properties data were subjected to ANOVA and the treatment means were compared using Tukey's Test of Honestly Significant Difference in SAS 9.3 (SAS Institute, Cary, NC, USA).

3. Results and discussion

3.1. Proximate composition of basundi mix

The moisture content of *basundi* mix was 2.9 ± 0.12% (d.b.), which was in the range of 2–4% reported for whole milk powder (WMP) [23]. Moisture content is an important property that directly affects the densities, reconstitution, shelf life and flow properties of powders. The fat content of the mix was 7.66 ± 0.41%, which was less than that of WMP (about 26%) but comparable to that of partial SMP. Such a low fat content was expected because the *basundi* mix is generally prepared using SMP as the base ingredient. In contrast, the protein content of 23.2 ± 0.33% in *basundi* mix was comparable to the value of 23.6–27.7% reported for WMP [24]. The ash content in *basundi* mix was 3.7 ± 0.19%, while the total carbohydrate content (62.54 ± 0.35%) was higher than that of WMP and SMP due to addition of sugar.

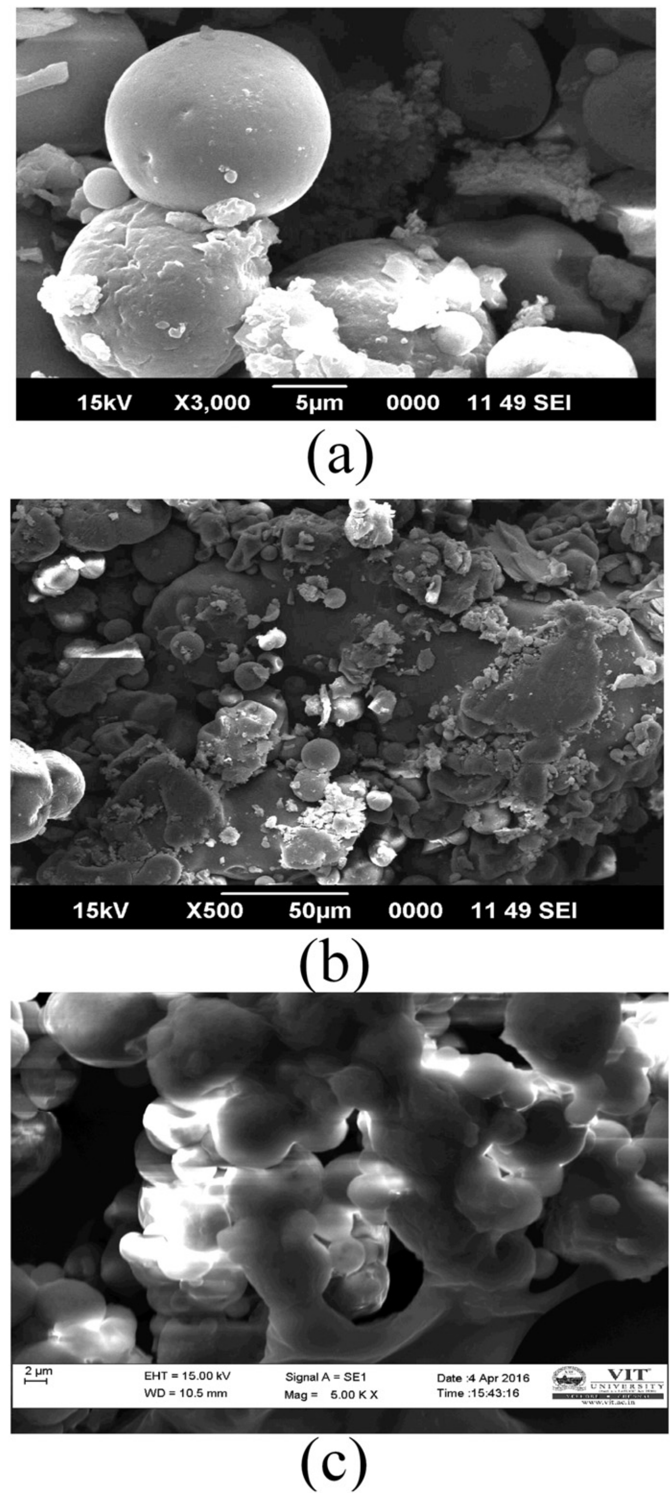


Fig. 2. Influence of moisture content on morphology of *basundi mix* (a) at 3% moisture content (3000×), (b) at 9% moisture content (500×) and (c) at 9% moisture content (5000×) resolutions.

3.2. Morphology

The SEM micrographs of *basundi* mix are given in Fig. 2. At 3% moisture content, the images revealed the spherical particles of milk and carbohydrates (starch or hydrocolloid). The sharp-edged particulates were that of sugar crystals. These sugar crystals solubilized at the higher moisture content of 9%, leading to the formation of lump-like structure. At 9% moisture content in the powder, the typical spherical milk particles were not evident and a lumpy mass was seen due to agglomeration of various particulates by water and sugar (Fig. 2b). The liquid bridging between particulates of *basundi* mix is evident in the SEM micrograph acquired at 5000× magnification (Fig. 2c).

3.3. Particle size distribution

The particle size distribution of *basundi* mix at different moisture contents is presented in Fig. 3. The mix demonstrated a smooth distribution of particle size in the range of moisture content tested. The mean $d_{0.1}$, $d_{0.5}$ and $d_{0.9}$ particle diameters at 3% moisture content increased from 21.91, 91.44 and 258.07 μm, respectively to 76.75, 292.47 and 446.77 μm at 9% moisture content. The $d_{0.1}$, $d_{0.5}$ and $d_{0.9}$ suggested that 10, 50 and 90% of the particles had diameter less than the respective value reported here.

As moisture content increased, the particle size distribution curve shifted to the right (Fig. 3). The influence of moisture content on the particle diameter was highly significant ($p < 0.01$). However, the shape of the volumetric particle size distribution curve remained unchanged. As the wet method of particle size determination was used, it could be inferred that the increase in particle was due to agglomeration and swelling of particulates in the presence of sugars. The values of span at 3, 6 and 9% moisture content were 2.58, 2.38 and 1.27, respectively. The lowest span at 9% moisture content was indicative of a narrow distribution in particle size.

3.4. Bulk and tapped densities

The bulk density of *basundi* mix ranged from 622.71 to 690.71 kg/m^3, and it decreased with increasing moisture content. Similarly, the tapped density decreased moderately from 819.10 to 729.31 kg/m^3 as moisture content increased from 3 to 9%. The effect of moisture content on bulk (ρ_{bulk}) and tapped densities is depicted in Fig. 4a. The tapped density was higher than bulk density because tapping enabled the smaller particles to occupy the voids between larger particulates and attain a dense packing condition. The changes in bulk and tapped densities with moisture content were pronounced ($p < 0.05$), suggesting the influence of strong inter-particle liquid bridges and interlocking forces between particles at higher moisture content. These liquid bridges and interlocking forces among particulates did not allow dense packing of

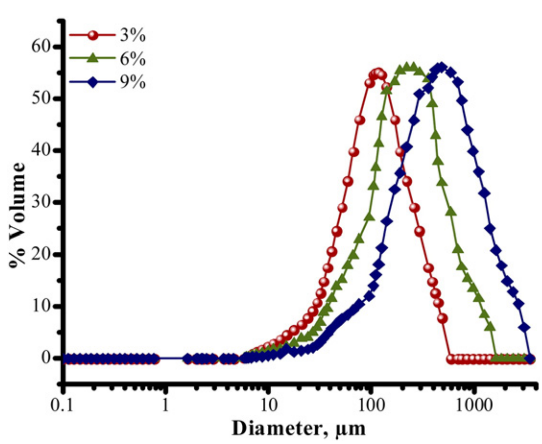


Fig. 3. Particle size distribution of *basundi* mix.

the powder during bulk and tapped density measurements. In contrast, the particle density was not significantly affected by moisture content ($p > 0.05$), and it lied between 1546.79 and 1567.90 kg/m^3.

As a consequence to the pronounced influence of moisture content on bulk and tapped densities, the porosity of the mix increased from 47.02% at 3% moisture content to 53.84% at 9% moisture content. The HR and CI of *basundi* mix were found to decrease significantly ($p < 0.05$) with change in moisture content from 3 to 9% (Fig. 4b and c, respectively). As particulates imbibed moisture and underwent swelling, the interparticulate forces acting on powder particles increased. In addition, solubilization of sugars at higher moisture contents also increased the strength of cohesive bonds between the particles. According to the powder flow classification proposed by Lebrun et al. [25] based on HR and CI values, *basundi* mix at 3 and 6% moisture contents was found to have better flowability as compared to that of 9% moisture content.

3.5. Angle of repose

The angle of repose of *basundi* mix decreased significantly from 41.49 to 36.96° as moisture content increased from 3 to 9% (Fig. 4d). Jha et al. [26] observed an angle of repose of 40.09° for instant *kheer* mix powder. As liquid bridges formed in the particulates with increasing moisture content, it was expected that the angle of repose would increase. Similarly, the presence of sugars in the product, and the induced cohesiveness and stickiness, should also have resulted in a higher angle of repose at higher moisture content. However, the particles agglomerated at higher moisture contents, and the size and sphericity of the agglomerates increased. The presence of sugars in the product also favoured agglomeration of solid particles. The larger agglomerated particles had relatively fewer inter-locking forces acting between particles, which favoured easy flow of the mix without forming a steep heap. Many studies in dairy powders showed that angle of repose and cohesion of powder particles were related to particle size, moisture content and chemical composition [27]. Quite similarly, it could be concluded that moisture content and presence of sugar were the prime factors that affected the angle of repose of *basundi* mix.

3.6. Moisture sorption behaviour

The MSI of *basundi* mix were largely sigmoidal in shape, characterized by two bending regions in the a_w ranges of 0.02–0.08 and 0.71–0.85 (Fig. 5). The moisture uptake was stable till 0.75 a_w, followed by an impulsive rise in the region III of the isotherm. The second bending of the isotherm was related to major shift in the binding pattern of water in the product. This shift could be reasoned to the presence of high amount of sugars in crystalline form in *basundi* mix, which became soluble at higher a_w. The GAB monolayer moisture content decreased from 2.291 to 1.063% as sorption temperature increased from 10 to 40 °C. Fig. 5 also showed that the EMC decreased with increasing temperature at constant a_w, crossover occurred at a_w above 0.66.

3.7. Glass transition temperature

The T_g of *basundi* mix as determined by DSC was found to be affected by moisture. It decreased steadily ($p < 0.01$) from 44.79 °C at 3% moisture content to 18.5 °C at 9% moisture content. The decrease in T_g of *basundi* mix with increase in free moisture was due to the plasticization effect of water. Such reduction in T_g is expected because water is an effective plasticizer for most biological materials. The exothermic reactions involving dissolution of sugars interfered with T_g.

3.8. Sticky point temperature

Stickiness or caking is a result of particle bridging in the presence of moisture. As expected, the SPT of *basundi* mix decreased linearly and

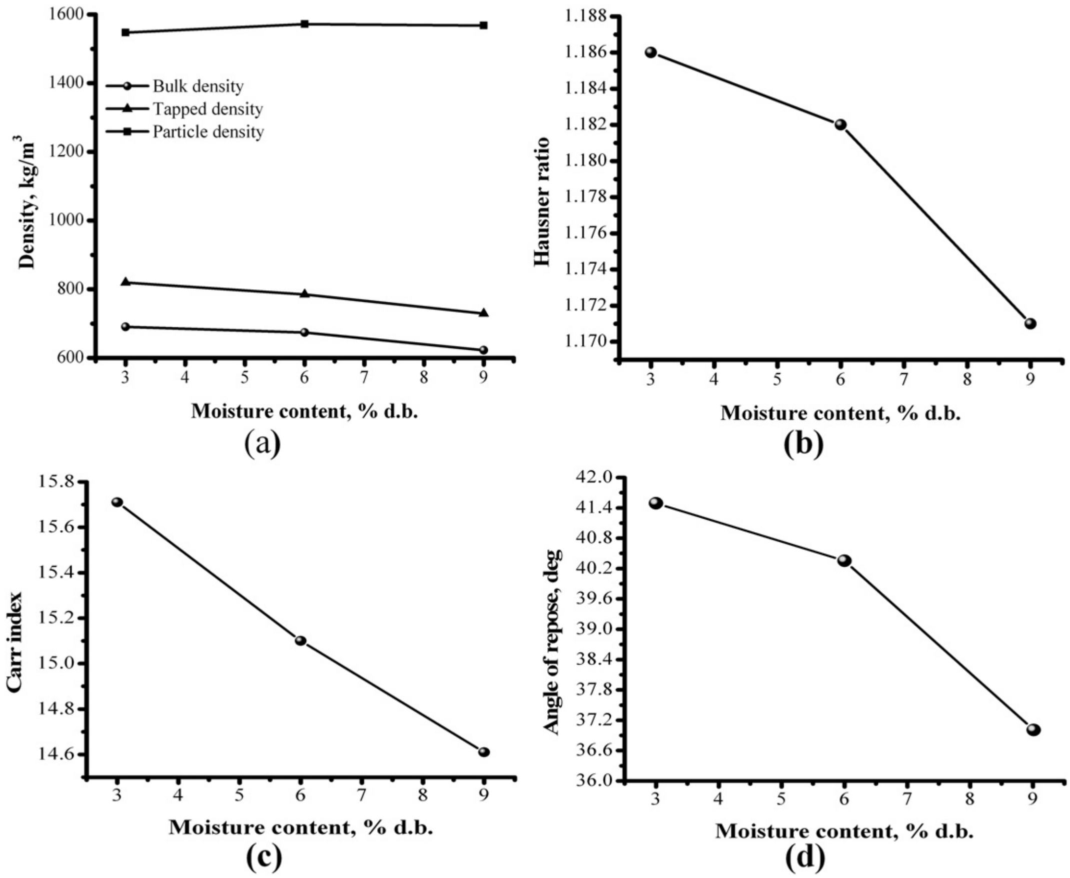


Fig. 4. Influence of moisture content on (a) Densities, (b) Hausner ratio, (c) Carr index and (d) angle of repose of *basundi* mix.

significantly ($p < 0.01$) with increasing moisture content, from approximately 40 °C at 2% moisture content to 10 °C at 8% moisture content. The moisture content at which stickiness was observed at a certain temperature was termed as the critical moisture content for caking to occur in the mix. The reduction in SPT could be attributed to the lowering of glass transition of sucrose, lactose, starch and the milk proteins, which were the major ingredients of this product. The experimental data of moisture content versus SPT fitted well to Eq. (13) as evident from the R^2 value of 0.97 and root mean squared error (RMSE) value of 1.77%.

$$SPT = -5.15 \times M + 49 \quad (13)$$

where 'M' was the moisture content of the product in dry basis. The decreasing trend in SPT at higher moisture contents was consistent with the findings of Hennings et al. [28] for SMP and Boonyai [29] for whey powder. From the SPT data, it could be concluded that *basundi* mix was very prone to caking even at ambient RH and temperature.

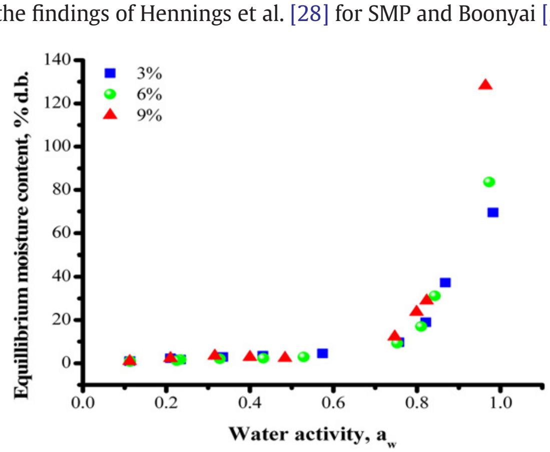


Fig. 5. Moisture sorption isotherms of *basundi* mix at different temperatures.

3.9. Influence of moisture content on dynamic flow properties

3.9.1. Basic flow properties

All powders are affected by moisture to some extent as the cohesive bonds and electrostatic charges that exist between particles are influenced by the level of moisture present. The dynamic flow properties of *basundi* mix as a function of moisture content are summarized in Fig. 6a-d. BFE, which is the energy required to displace a conditioned powder sample [6], increased with increase in moisture content (Fig. 6a). BFE measurement is often the most revealing parameter as it can detect even very small variations in the physical properties of different samples such as particle size distribution, particle shape, surface roughness, moisture content, and electrostatic charges. The agglomerated larger-sized particles at 9% moisture content needed significantly ($p < 0.01$) more energy to move as compared to 3% moisture content powder in the forced flow regime. Bian et al. [30] observed the BFE value of 680 mJ for hard wheat and 713 mJ for soft wheat flour at 11.4% moisture content, which was much lower than the BFE observed for *basundi* mix. Similarly, BFE ranging from 127 mJ to 157 mJ have been reported for SMP, demineralised whey powder and whey powders [31]. BFE variations could be complex to interpret as higher BFE could be indicative of either more free-flowing or less free-flowing powder.

A possible explanation for the behaviour was that for cohesive powders the flowing zone ahead of and around the blade in which shearing occurred was extensive and there was high transmissibility of forces from particle to particle throughout the system. Therefore, a high proportion of sample volume was moved, and a kind of chain reaction took place, as the blade penetrated the powder. Thus, more

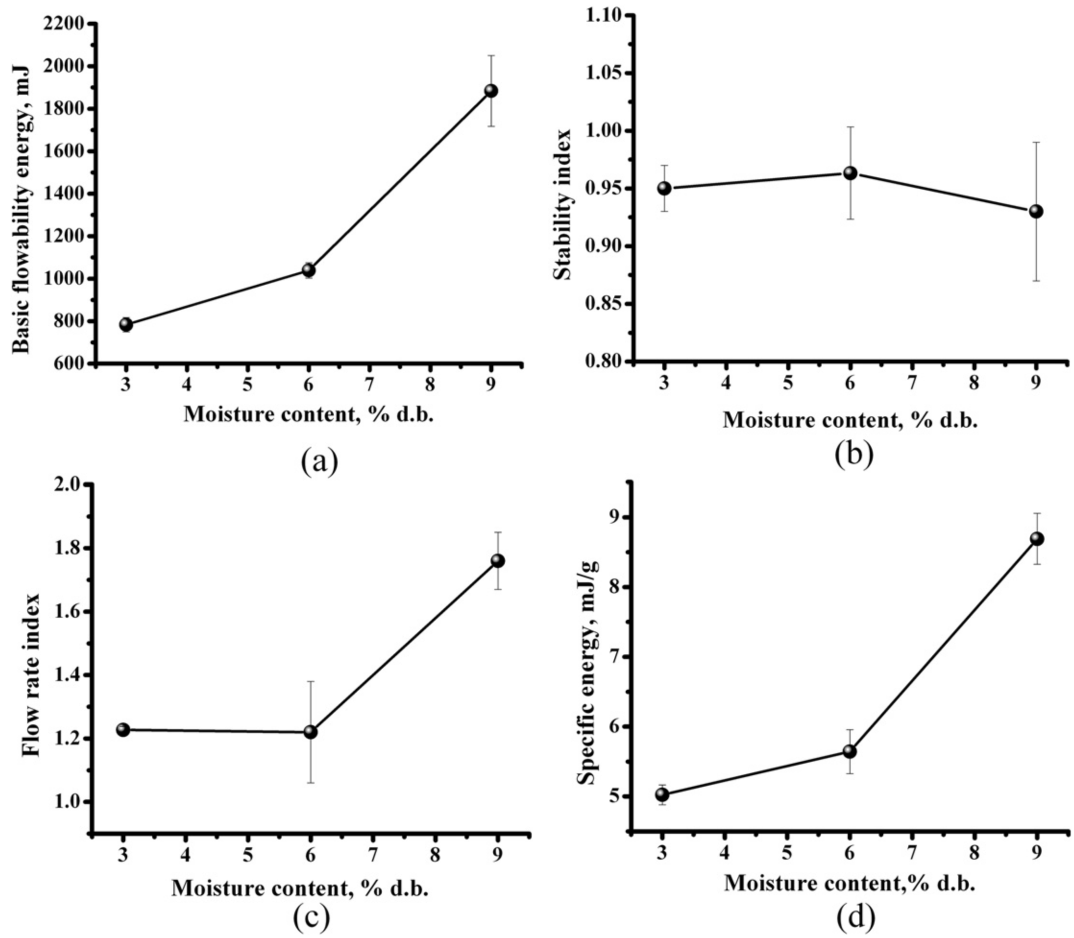


Fig. 6. Influence of moisture content on (a) basic flowability energy, (b) flow rate index, (c) stability index and (d) specific energy of *basundi* mix.

work was done in the case for a cohesive powder. On the contrary, the flow zone of non-cohesive materials ahead of and around the blade as it moved was much smaller so that the number of particles that were sheared or moved relative to the nearby particles was smaller. Therefore, the work done for the same volume would become low at lower moisture content.

The SI value indicates whether the powder is changing during flow testing. *Basundi* mix had SI in the normal range of 0.9–1.1 (Fig. 6b) for flow, as defined by Bian et al. [30]. Above or below this range, food powders are prone to attrition and would be categorized as unstable. Instability may occur when powders are very cohesive and compressible, may become caked or agglomerated during flow, thereby changing the flow properties significantly. Increase in moisture content exhibited a negligible effect ($p > 0.05$) on the SI of *basundi* mix.

The FRI measures how sensitive the powder is to being made to flow at different rates, by varying the blade tip speed. The FRI of *basundi* mix was closer to 1.2 (Fig. 6c) at 3% and 6% moisture content, indicating the powder was insensitive to varying flow rate ($p > 0.05$). However, the FRI at 9% moisture content indicated that the mix possessed average flow rate sensitiveness and the value was considerably higher (1.79). The cohesive powders required greater flow energy at lower flow rates because the entrained air was able to escape, leaving a stiffer material which was more resistant to flow. The non-cohesive powders were less sensitive to flow rate because of the presence of entrained air.

The SE increased significantly ($p < 0.05$) with increase in moisture content of the powder. The mix was cohesive at 9% moisture content with higher SE value as compared to those values at 3 and 6% moisture contents. The SE was more dependent on the cohesive and mechanical interlocking forces between the particles, and less influenced by other factors such as compressibility. MSI revealed dissolution of sugars at RH above 0.7, which caused the stickiness and cohesiveness in the powder owing to the formation of strong liquid bridges (Fig. 5). The isotherms also revealed a major shift in water binding only at 9% moisture content. SEM micrographs showed the presence of such liquid bridges in the powder at 9% moisture content, corresponding to a_w of 0.75 (Fig. 2c). The SE value at 3% moisture content was lower than that at 9% moisture content (Fig. 6d), indicating that it was the least cohesive sample in an unconfined flow regime, and was consistent with relatively low BFE value. As the probe traversed through the particulate sample, the intermolecular forces between the particles acted on the blade during its upward movement and there were no other forces constraining the top layer of the powder. SE values between 5 and 10 mJ/g indicated that *basundi* mix was moderately cohesive. In summary, a higher BFE value (lower flowability) and higher SE value (higher cohesivity) of 9% moisture *basundi* mix as compared to 3% showed that it was a more stable sample (and less flowing) in this flow regime. The major shift in water binding characteristics and the dissolution of sugars with increase in moisture content from 6 to 9% caused large charges in the dynamic flow properties as compared to the changes in the moisture content region of 3 to 6% (Fig. 6a-d). The transition of sugars from crystalline to rubbery state was one of the reasons for such drastic changes in flow behaviour, which made flow of *basundi* mix difficult.

3.9.2. Influence of moisture content on compressibility

The compressibility test is a bulk property measurement that reveals how density changed as a function of applied normal stress. Though compressibility is not a direct measure of flowability, it can be used to understand the behaviour of powders in various process or handling environments. Free-flowing powders tend to be less sensitive to direct compression. *Basundi* mix showed high compression value of 17% at 6% moisture content followed by 15.9% at 3% moisture content and 13.2% at 9% moisture content (Fig. 7a). As the particle size was smaller at 3 and 6% moisture contents, it favoured compression. The compressibility of *basundi* mix at 9% moisture content was the least because the large-size particulates and the presence of strong liquid bridges formed by solubilizing sugars (Fig. 2c) resisted compression. Similar influence of moisture content on compressibilities was reported by Manikantan et al. [32] in coconut flours. The compressibility at 3% moisture content was marginally lower than at 6% moisture content because at the lower range of moisture sufficient water was not available for cohesion of the particles, which is also requirement for compressibility.

3.9.3. Influence of moisture content on aeration energy

Basundi mix at all three moisture contents was sensitive ($p < 0.05$) to low levels of aeration. The data obtained from this study showed that the least amount of energy was required to aerate *basundi* mix at lower moisture content (3%) as compared to that at higher moisture contents. Fig. 7b indicated that aerating the bed increased the flowability of *basundi* mix as the total energy required decreased drastically with decrease in moisture content. This was because air decreased the interparticulate cohesive forces and induced flow at a lower energy. Fig. 7b also indicated that a minimum air velocity of 10 mm/s was required to break the inter-particular forces. The mix at 9% moisture content was slightly more sensitive to higher levels of aeration as compared to 3 and 6%. However, *basundi* mix at 3% moisture content had the lowest aeration energy, indicating overall lower cohesivity in the aerated environment.

3.9.4. Influence of moisture content on shear properties

The shear test provides an indication of how easily a powder will move from a static condition to dynamic flow. Thus, shear properties provide information on whether the powder will flow or bridging, blockages, and stoppages are likely to occur. The shear parameters such as AIF and ff_c decreased from 50.41 to 38.86° and 4.55 to 2.37, respectively with increase in moisture content (Fig. 8a-d) while wall friction and cohesion increased. A larger ff_c was suggestive of a smaller ratio of UYS to consolidation stress, and better the powder would flow. According to the classification proposed by Jenike [33], a powder is cohesive if its flow function ranges between 2 and 4 and very cohesive between 1 and 2. Thus, the flow behaviour of *basundi* mix could be classified as "cohesive" at 3% moisture content to "very cohesive" at 9% moisture content. The UYS decreased from 9.16 to 7.41 kPa as moisture increased from 3 to 9% (figure not shown). For comparison, the UYS of demineralised whey, SMP and whey powders were reported as 1.37, 4.48 and 4.69 kPa, respectively [31].

In contrast, there was a steady increase in the cohesion scores, which was due to increase in wetting. The cohesion values obtained for *basundi* mix increased markedly from 5.46 kPa at 3% moisture content to 13.94 kPa at 9% moisture content (Fig. 8a). The higher UYS and cohesion values at 9% moisture content suggested that the mix became very cohesive as moisture increased [34]. Correlating SEM micrographs and moisture sorption data, it was evident that adsorption of moisture above 6% encouraged particle to particle interaction due to liquid bridging and interlocking forces between powder particles [35]. The liquid bridges were very probably strong due to the dissolution of sugars in water and the increase in viscosity (Figs. 2c and 5), which also increased cohesion on the surface of powder particles in addition to the effect of moisture. The drastic reduction in T_g and SPT with increase in moisture content from 3 to 9% is an affirmation to the increase in cohesiveness. At higher moisture content, the bridging forces between particles were presumed to have dominated the van der Waals forces [36].

Higher the AIF, the more difficult it is for the powder to move along the wall surface. It is the measure of the internal friction at steady-state flow of granular materials. AIF increased from 50.1 to 53.4° with increase in moisture content from 3 to 6% ($p > 0.05$), but decreased substantially ($p < 0.05$) to 38.5° at 9% moisture content. It is well-known that powder flow properties are dependent on particle size and its distribution [37]. Lapčík et al. [31] reported frictional angles of 26.5, 36.4 and 40.4° for whey, demineralised whey and skimmed milk powders, respectively. The flow function value was similar to that of the AIF, and it decreased with increasing moisture content (Fig. 8c). The results obtained were in accordance with those of Fitzpatrick et al. [38], who suggested that lower flow function values (towards the bottom of the graph) represented easy flow of materials.

3.9.5. Influence of moisture content on wall friction

Wall friction results help in assessing the frictional resistance offered by the bin/hopper wall material on the flow of powder materials. The calculated wall friction angles of *basundi* mix were 29.60°, 32.43° and 38.06° for 3, 6 and 9% moisture contents, respectively. The influence of moisture on wall friction was slightly different ($p < 0.05$) from that of angle of repose. As the sugars dissolved at higher moisture content, the plasticizing effect of water and sugar exerted strong cohesion with the metal surface. The influence of moisture content of *basundi* mix on wall friction was remarkable as observed from Fig. 8d. The values of wall friction were in accordance with increasing cohesivity, which in

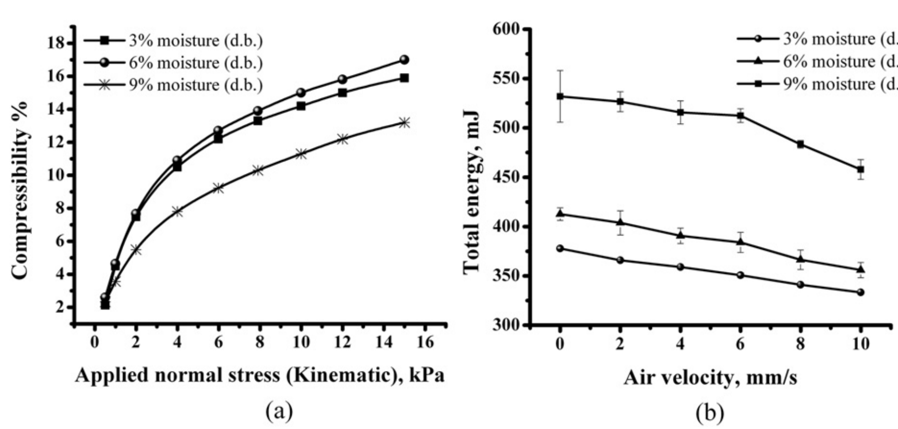


Fig. 7. Influence of moisture content on (a) compressibility and (b) aeration energy of *basundi* mix.

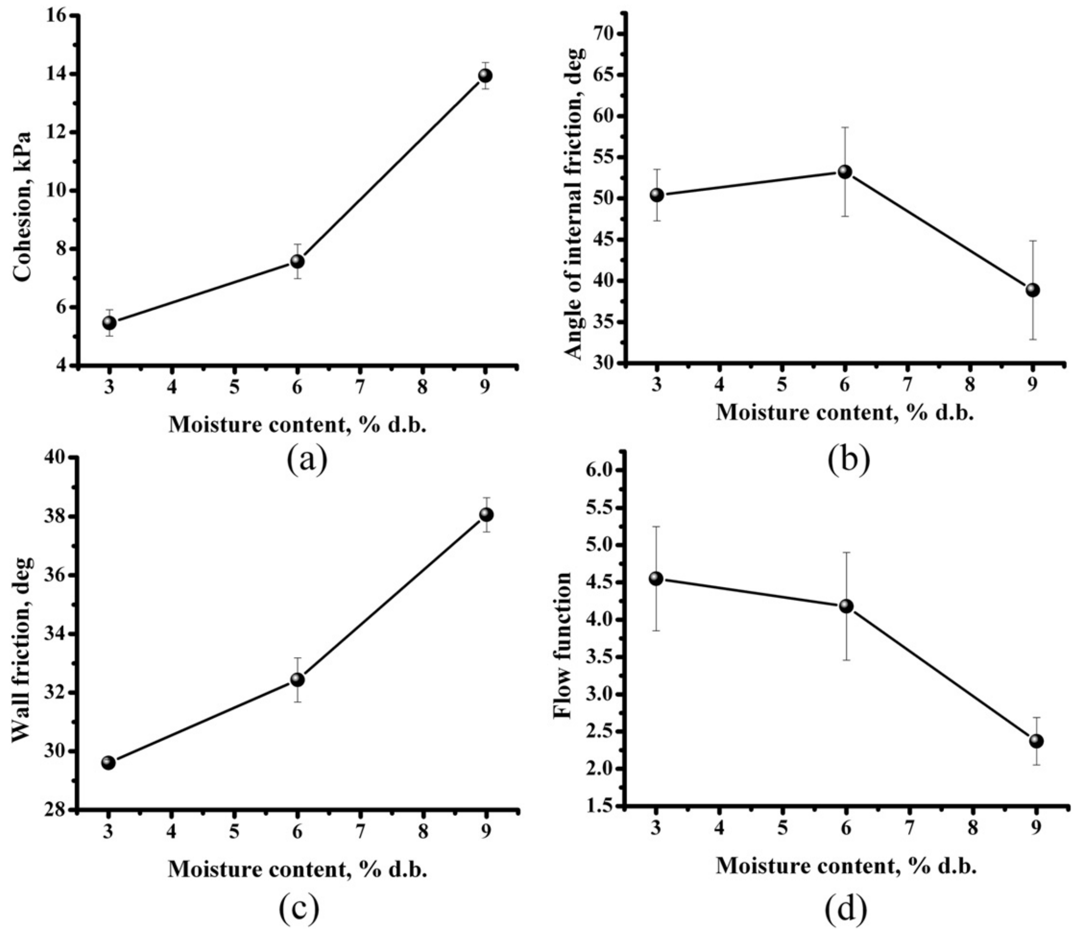


Fig. 8. Influence of moisture content on (a) cohesion, (b) angle of internal friction, (c) flow function and (d) wall friction angle of *basundi* mix.

turn was related to moisture content. The values obtained also indicated that hopper sections should be the steepest for 9% and the gentlest for 3% moisture *basundi* mix.

3.9.6. Optimal conditions of storage of basundi mix

The basic flow and shear properties data suggested that *basundi* mix had the best flowability at 3% moisture content. The moisture content corresponding to SPT of 30 °C was 3.6% (Eq. (13)). From the moisture sorption data, it was evident that the a_w range for the moisture content range of 3–3.6% was 0.3–0.4. Thus, correlating the SPT, flow properties and moisture sorption data, the recommended optimal conditions for storage of this product would be 30 °C temperature and 40% RH. Beyond these conditions, *basundi* mix could become sticky and might not flow well.

4. Conclusions

The study analyzed and differentiated the flow-related properties of *basundi* mix at different moisture contents. The test results demonstrated that the flow-related properties of the mix were largely affected by moisture content. Such differences in the flow properties occurred because of agglomeration of powder particulates that changed interparticular behaviour during flow. Packing of smaller particles in void spaces and higher cohesion resulted in higher aeration ratio. The T_g and SPT data also suggested that *basundi* mix would be very cohesive and susceptible to caking. The major shift in water binding characteristics due to adsorption of water and dissolution of sugars in the moisture content range of 6 to 9% caused large charges in the basic flow properties. Shear tests showed that *basundi* mix was relatively more flowable at 3% moisture content. Shear properties also indicated that *basundi* mix was highly cohesive at higher moisture content and would require a steeper hopper design in a storage bin to necessitate mass flow. The results of static flow behaviour (angle of repose) and the dynamic flow tests contrasted each other. Thus, it could be stated that dynamic tests are better in characterizing flowability of powders, particularly those containing sugars. This study evaluated the flow properties that affect the behaviour of *basundi* mix during processing, handling and storage, and was useful in optimizing its storage conditions.

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