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Freeze drying of ash gourd juice with carrier agents and evaluation of physicochemical and techno-functional properties

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Abstract

This study was undertaken to develop ash gourd juice powder (AGP) with the assistance of different carrier agents *viz.*, maltodextrin (MD), gum arabic (GA), soy protein isolate (SPI) in different proportions by freeze-drying. The carrier agents were added in 1:10, 3:20 and 1:5 (w/v) to study their effect on the physicochemical properties like moisture content, color and techno-functional properties like powder recovery, bulk density, tapped density, Hausner ratio, Carr index, swelling capacity, water and oil holding capacity, water solubility index and hygroscopicity. GA as carrier agent recorded the highest yield (16.04%) followed by samples with SPI and MD as carrier agents. AGP produced with maltodextrin (1:5) showed more lightness (L*) than MD (1:10) and MD (3:20) whereas, GA based sample at 1:5 showed more lightness (L*) than SPI (1:5). AGP samples with SPI as carrier agent showed higher moisture content, bulk density and tapped density (TD) compared to GA and MD added samples. Also, the moisture content, bulk density and tapped density of dried powders decreased with an increase in carrier agents proportion. Dried AGP obtained from MD as carrier agent showed more oil holding capacity and the sample with a carrier agent showed better water holding capacity. Higher swelling capacity was observed in samples with MD (1:5) as a carrier agent than (1:10) and (3:20). Powder with MD as a carrier agent showed higher water solubility index (WSI%) with a trend of increase in carrier agent proportion. However, hygroscopicity (HY%) was higher in samples with MD compared to samples with SPI and GA. Also, the HY% decreased with an increase in carrier agent proportions. The wettability of AGP decreased with an increase in carrier agent proportion. Powder obtained through GA as a carrier agent took more time than other carrier agents like SPI and MD. Overall, the study indicated that ash gourd can be dried by using freeze dryer to protect techno-functional and physicochemical properties. Further, the nutritive composition of the samples needs to be assessed to evaluate the change in composition after freeze drying.

Key words: Ash gourd (*Benincasa hispida*), juice, powder, freeze drying, moisture content, carrier agent

Introduction

Ash gourd (*Benincasa hispida*) belongs to cucurbitaceae family having medicinal and functional properties. It derives the name due to the color of the skin which resembles ash. In few geographical locations, the skin of gourd is waxy and shine hence, it is also called as wax gourd. Ash gourd is predominantly cultivated in India, China, Indonesia, Taiwan and Japan. Deep loamy soil with the pH range of 5-7.5 is suitable and a humid tropical climate is ideal for increased productivity. The average productivity of ash gourd in India is reported as 75-85 tonnes per acre. Kerala is one of the leading states in cultivation of ash gourd, Andhra Pradesh, Karnataka and Punjab cultivates this gourd in limited areas.

It is reported to be rich in micro nutrients like Ca, Mg, Fe, Cu, Zn and Se. The pulp, leaves, seeds and flowers have different functional and therapeutic properties. In Kerala, it is one of the principal ingredients in preparation of ayurvedic medicine *Kushmanda Rasayana*. Musale *et al.* (2017) reported that the nutritional value per 100 g of ash gourd is moisture (96.5%), protein (0.4 g), fat (0.1 g), carbohydrate (1.9 g), ascorbic acid (1 mg), fiber (0.8 g), ash (0.39 g), iron (0.8 mg), calcium (30 mg). It exhibits low calorific value of 10 kcal from edible portion. Total dietary fiber in ash gourd is beneficial in lowering blood cholesterol level and mitigates incidence of coronary heart diseases and bowel disorders (Palamthodi *et al.*, 2019). Low carbohydrate content of ash gourd makes it a good source of diet for diabetic and hypertensive patients (Bello *et al.*, 2014). The vitamins in ash gourd assist in curing common cold, flu, pneumonia, and migraine.The taste of ash gourd is mild like cucumber with typical aroma leading to discontent by consumer.

Drying is one of the preservation techniques used for perishable products by removing moisture using various medium. Freeze drying is one of the drying techniques, where moisture is removed by sublimation facilitating low drying temperature conditions. The drying technique involves pre- cooling, phase change and solidification stage (Dalvi-Isfahan *et al.*, 2017). Color, texture, odour, rehydration, water activity, flow property, bulk density properties, retention of volatile and nutritive compounds are influenced by drying process. Freeze drying of vegetables are reported to have retained ascorbic acid, carotene, phenol compounds and aroma across many groups compared to conventional drying techniques (Bhatta *et al.*, 2020).

The objective of producing powder for better retention of compositional ingredients greatly depends on process parameters in drying. In case of freeze drying, the product is exposed to negetive temperatures under low pressure conditions. Also, it is expensive drying technology due to energy requirement for operation of pump, refrigeration and vacuum creation in drying chamber. The carrier agents used to avoid sticky nature of powder was maltodextrin, gum arabic, waxy starch, pectin, vegetable fibers, and starches (Adhikari *et al.*, 2004; Osorio *et al.*, 2011; Wang and Zhou, 2012 and Sablani *et al.*, 2008). Gum Arabic, maltodextrin and modified starch (Yamashitha *et al.*, 2017) were tried as encapsulating material for freeze drying.Ash gourd consumption is very limited owing to its non-palatability to the consumer. Due to market volatility, there is a glut in ash gourd production leading to distress sale by farmers subsequently leading to post-harvest losses. To increase palatability, it can be converted into powder and incorporate into suitable food products. Studies were conducted for conversion of juices derived from vegetable and fruits into powder by spray and freeze dryer. Influence of different carrier agents on functional properties of powders was also reported. However, no work is reported on freeze drying of ash gourd juice by using maltodextrin (MD), soy protein isolate (SPI) and gum Arabic (GA). Hence, the present study was conducted with the following objectives. (i) Freeze drying of ash gourd juice with MD, SPI and GA as carrier agents at different proportions. (ii) Evaluation of physicochemical and techno-functional properties of developed powder.

Materials and methods

Raw materials and equipment: Matured ash gourds were procured from local market of Pulivendula town of Kadapa dist., Andhra Pradesh. Gourds were freshly harvested (1 day old) and handled with care during its usage in all experiments. Edible grade MD, SPI and GA were procured from authorized suppliers. Chemicals used in the experiments were of analytical grade and were procured from reputed firms.

Lab scale bench top freeze dryer with an ice capacity of 1.5 kg was used in the present study. MiniLyodel (Delvac Pumps Pvt. Ltd., Thirumudivakkam, Chennai, Tamil Nadu, India) which is ideal for freeze-drying applications was used for all experiments, also having 8-port column flask manifolds for drying of liquid material.

Preparation of ash gourd juice: Freshly procured ash gourd was thoroughly washed using tap water and surface moisture was removed by wiping with dry cloth. Gourd was cut into pieces to remove peel from white color pulp. Pulp was diced into cubes with stainless steel knife followed by crushing using domestic mixer by adding known volume of water. Crushed juice of ash gourd was strained through four-fold muslin cloth and stored in refrigerated condition.

Freeze drying of juice using carrier agents: Trials conducted without addition of carrier agents resulted in sticky powder which was difficult to recover from freeze plates. Hence, carrier agents were added to juice obtained in above step for improving powder extraction. Through trails, proportion of carrier agents to be added to ash gourd juice was selected as 1:10, 3:20 and 5:10 on weight to volume ratio. Ash gourd juice (AGJ) obtained in the previous step was mixed with edible grade MD, SPI, GA in three proportions. Thoroughly mixed juice was poured into freezer plates in thin layers and loaded into deep freezer for pre-freezing of sample. Solidified mass of AGJ in plates were transferred to drying chamber platform of freeze dryer. Chamber was closed with enclosure provided with gaskets to create air tight conditions. Refrigeration unit of the equipment was put on to bring down temperature of freezing chamber to -40°C. After attaining this temperature, vacuum unit of the chamber was put on and maintained at (<1mm) till completion of freeze drying (24 h).

Evaluation of techno-functional properties: Powder recovery of AGP was calculated by the method described by Bhat *et al.* (2021). Recovery of powder was calculated as percentage recovery of total solids in feed to that of total solids in recovered powder. Powder recovery was calculated by the following equation:

$$\text{Powder recovery}(\%) = \frac{\text{Amount of powder obtained after drying}}{\text{Amount of feed to dryer}} \times 100 \quad (1)$$

Bulk and tapped density was calculated using method followed by Premi and Sharma (2017). Accurately 2g of AGP was freely poured into a 10 mL graduated glass cylinder. The bulk density (BD) of samples was calculated as ratio of mass of powder and volume and expressed as g/mL.For calculation of tapped density, AGP was loaded into measuring cylinder and tapped 10 times from a distance of 10 cm and resultant density was called as tapped density.

$$\text{Bulk density} = \frac{\text{Mass of the powder (g)}}{\text{Volume occuped in the cylinder (mL)}} \quad (2)$$

Flowability and cohesiveness of AGP was estimated as Hausner ratio which classify powders into various groups. It is the ratio of tapped density to bulk density and the resultant values are grouped as follows. $1.0 < HR < 1.1$, free-flowing powder; $1.1 < HR < 1.25$, medium-flowing powder; $1.25 < HR < 1.40$, difficult flowing powder; $HR < 1.4$, very difficult flowing powder.

The flow properties of solids were categorized into different flowability groups based on tapped and bulk density, as described by Carr, R.L. According to Carr's classification, a Carr index (CrI) in the range of 5–15% indicates excellent flowability, whereas CrI values greater than 25% suggest poor flowability. The Carr index (CrI) of AJP was calculated using the following equation:

$$\text{Carr index (CrI)} = \frac{\rho_{tapped} - \rho_{bulk}}{\rho_{tapped}} \quad (3)$$

Swelling index was determined according to the method described by Onwuka and Onwuka (2005) with slight modifications. AGP was filled into 50mL measuring cylinder till 5mL mark. Distilled water was gently added up to 25 mL mark. The contents were tightly covered and mixed by inverting it several times for 2 min. Cylinder was allowed to stand for 5 min. to measure the volume occupied by the sample. Swelling capacity was calculated using formula.

$$\text{Swelling capacity} = \frac{\text{Volume occupied by sample after swelling}}{\text{Volume occupied by sample before swelling}} \quad (4)$$

Water and oil holding capacity of AGP was estimated as per method reported by Beuhat (1977). AGP (0.5 g) was mixed with 10mL of distilled water/edible oil in centrifuge tubes and vortexed for 30s. The dispersions were allowed to stand at room temperature for 30 min and centrifuged at 3000 rpm 25 min. The supernatant was decanted into measuring cylinder and volume occupied was noted. The difference between initial volumes of distilled water/edible oil added to the sample and the volume obtained after centrifugation was noted. Oil and water absorption for values were reported as mL of water absorbed per gram of sample.

For estimating water solubility index, AGP (1g) was mixed with 10 mL of distilled water in a centrifuge tube. The mixture was incubated at 37°C for 30 min. in a water bath followed by centrifugation at 5000 rpm for 20 min. The supernatant was carefully decanted into a pre-weighed petri dish and dried in a hot air oven at 105°C for 4 h (Hogekamp and Schubert, 2003).

$$\text{WHC/OHC} = \frac{\text{Amount of water absorbed}}{\text{Weight of sample}} \quad (5)$$

Hygroscopicity of AGP samples was evaluated as per the method described by Cai and Corke (2000) to know hygroscopicity. One gram of AGP was spread on petridish and placed in an airtight plastic container filed with Na_2So_4 saturated solution (81% RH). Sample was weighted after one week and hygroscopicity of sample was expressed as g of moisture per 100 g dry solids (g/100 g).

$$\text{Hygroscopicity (\%)} = \frac{\text{Weight of absorbed moisture}}{\text{Weight of dry solid}} \times 100 \quad (6)$$

Evaluation of physico- chemical properties: Physcio-chemical properties of powders determine the shelf stability of powders in addition to its intrinsic quality. A thorough investigation of the physico-chemical properties of AGP was conducted to evaluate its potential as an ingredient in conventional food products. This analysis also aimed to understand its chemical nature and its possible interactions and impact on co-ingredients in food formulations.

Hunter lab colorimeter was used to measure the color of the AGP samples. White and black plates were used for the calibration of equipment as per instructions prescribed in user manual. AGP colour values were measured in terms of L*, a* and b*where, L* indicates lightness (100) and darkness (0). The other two coordinates a* and b* represents redness (+a* value) to greenness (-a* value) and yellowness (+b* value) to blueness (-b* value).

Moisture content of AGP samples were measured as per AOAC (2000) protocol. Two grams of AGP was taken into a teared petri dish. Samples were dried in a hot air oven at 105°C till constant moisture content was obtained. Moisture content (MC) of the sample was calculated as:

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (7)$$

W_1 = Weight (g) of sample before drying; W_2 = Weight (g) of sample after drying

Results and discussion

All experiments were conducted in triplicates and their mean value was analysed for interpretation of results. For simplification, the treatments with maltodextrin with 1:10, 3:20 and 1:5 are indicated as MD1, MD2 and MD3. Similarly, for soy protein isolate SPI1, SPI2 and SPI3, respectively for three ratios. Whereas, gum Arabic with GA1, GA2 and GA3.

Powder recovery: Recovery of powder from drying process is influenced by carrier agents. Powder yield from carrier agents in different ratios is depicted in Fig. 1. Amongst the carrier agents, gum arabica resulted in better powder yield compared to remaining two carrier agents. Also, powder yield improved with increase in carrier agent proportion across all groups.

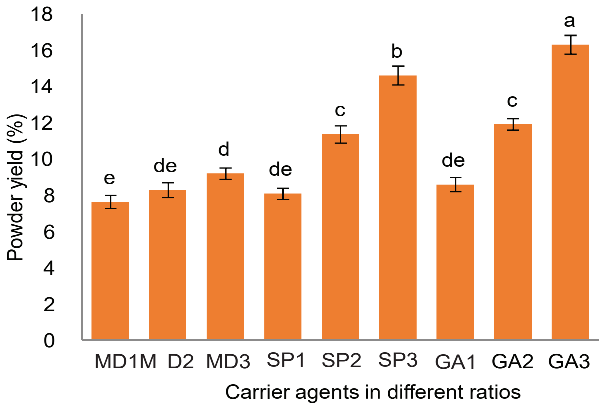

Fig. 1. Ash guard yield from freeze drying using carrier agents at different ratios. Different alphabets in bar graph apex indicate significant different at (P<0.05) by Tukey's test)

Increase in carrier agent proportion resulted in higher recovery might be due to binding effect of carrier agent with AGJ. Free moisture in juice was binded by carrier agent and in turn resulted in better powder recovery during freeze drying. During experimentation, it was observed that GA samples were porous and easily flowable compared to other samples. Kalušević *et al.* (2017) reported that GA in combination with soybean coat extract reported yield of 63.7% by spray drying. However, in the study MD reported better results compared to GA contradicting our findings.

Bulk and tapped density: These two characteristics play an important role in handing of powders and storage. Powders with suitable bulk and tapped density value are easy to handle and mix with other ingredients. Highest bulk density was reported in SPI and least value in MD samples (Table 1). However, there was no significant difference between different proportion of MD(P<0.05). Tapped density was also highest in SPI and least value was reported in MD samples. Higher bulk density indicates lower void space and more material weight. With increase in carrier agents, the bulk density and tapped density increased owing to influence of carrier agents' presence in resultant dried powder. While evaluating physical properties of milk powder Pugliese *et al.* (2017) reported average bulk density of 541.36 kg/m^3.

Hausner ratio: The Hausner ratio is a measure of frictional conditions between powder particles. It was shown in studies of different powder samples that the increase of particle size

Table 1. Bulk and tapped density of ash guard powder from carrier material of different ratio

Sample	Bulk Density (g/mL)	Tapped Density (g/mL)
MD1	0.307 ± 0.010^{e}	0.570 ± 0.009^{e}
MD2	0.453 ± 0.006^{e}	0.594 ± 0.594^{e}
MD3	0.453 ± 0.006^{e}	0.674 ± 0.005^{d}
SPI1	0.553 ± 0.021^{d}	0.665 ± 0.028^{d}
SPI2	0.593 ± 0.031^{cd}	0.730 ± 0.045^{cd}
SPI3	0.762 ± 0.021^{a}	0.876 ± 0.017^{a}
GA1	0.557 ± 0.021^{d}	0.723 ± 0.025^{cd}
GA2	0.630 ± 0.010^{bc}	0.786 ± 0.025^{bc}
GA3	0.673 ± 0.005^{b}	0.847 ± 0.020^{ab}

Different alphabets subscripted in each column indicate significant difference (P<0.05) by Tukey's test improves flowability. The increase in mass of individual particles leads to an increase in the relative influence of gravitational forces over inter particle forces, resulting in this improvement. Fig. 2 shows the Hausner ratio values for different samples.

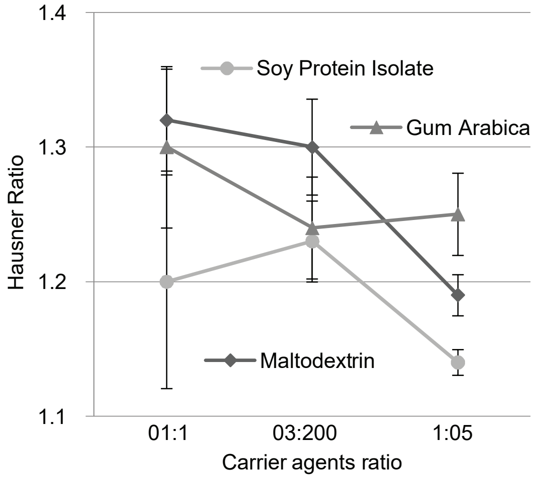

Fig. 2. Hausner ratio of ash gourd powder with carrier agents of different ratios

There was no particular trend observed in the samples with different carrier agents. It ranged between 1.323 and 1.149 across different samples with samples added with MD reported with highest value. Excluding sample SPI3 and MD3, remaining samples are categorized as difficult to flow category. This behaviour of powder can be attributed to influence of carrier agents and drying process wherein samples are in fine particulate size. Carrier agents in higher proportion helped in better moisture removal and aggregation of small particles into larger size could be attributed to this trend.

Carr index: Commonly used measure to understand the particulate interactions is the compressibility index which is often referred as Carr's index. MD at lower proportion exhibited highest Carr's index whereas, SPI reported least value. Excluding four samples *viz.*, MD1, MD2, GA1 and GA3, remaining samples reported carr index less than 20% which emphasizes free flowability of powder. Samples with SPI as carrier agent reported less than 20% values across all ratios. However, there was no trend with ratio of carrier agents added to AGP. It is reported that Carr index is independent of moisture ratio. However, Nokhodchis and Rubinstein (2001) reported that moisture content had a significant effect on the compressibility of hydroxypropyl methyl cellulose. On the other hand, lower Carr index indicate lower cohesive nature of powder. Sahni and Shere (2017) reported Carr index of 19.64, 21.56 and 23.07% for apple pomace, carrot pomace and beetroot pomace powder, respectively obtained by tray drying.

Swelling capacity: Swelling capacity is an indicator which denotes the degree of granule hydration in sample. High swelling capacity is an indication of weaker binding forces in the granules. Also, it is reported that increase in temperature results in weaker binding interactions between molecules and hence the enlargement in chains that allows higher entrapment of water molecules. Fig. 3 indicates swelling capacity of ash gourd powder obtained by carrier agents in different proportions.

Swelling capacity of ash gourd samples varied from 1.2 to 1.761 across different carrier agents. Swelling capacity increased with

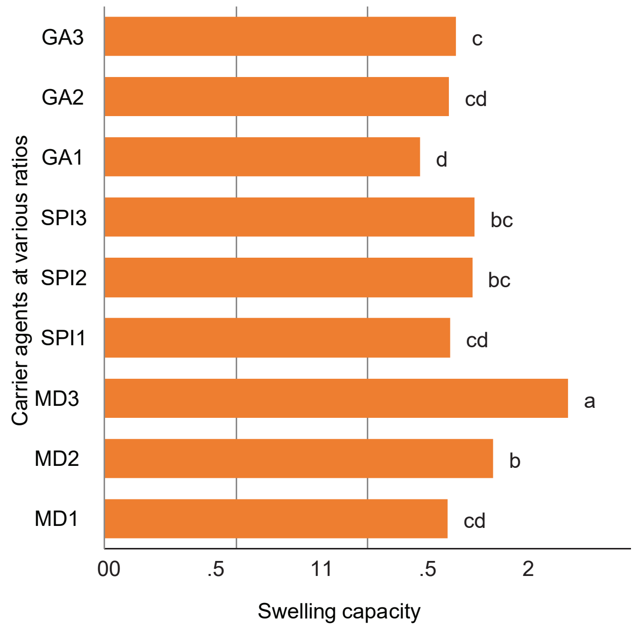

Fig. 3. Swelling capacity of ash gourd powder with carrier agents of different ratios. Different alphabets in bar graph apex indicate significant different at P<0.05 by Tukey's test

increase in carrier agent proportion across all types. Among the carrier agents, samples with MD reported highest value for swelling capacity with 1.761 at 1:05 carrier agents. During course of freeze drying with various carrier agents, the structure of powder was maintained puffy and the same was regained after rehydration. Ogechukwu and Ikechukwu (2017) experimented lime bean flour to investigate heat processing impact on functional properties. The study reported values ranging from 1.48 to 1.68 which is on par with values reported in the present study.

Water and oil holding capacity: Water-holding capacity (WHC) is an important property wherein protein–water interaction occurs in various food system influencing functional properties of foods. It also indicates the ability of protein matrix to absorb and retain bound, hydrodynamic, physically entrapped and capillary water against gravity. OHC is related to surface properties of fiber particle, overall charge density and polysaccharides hydrophilicity of components.

MD added samples showed higher OHC compared to other two carrier agents. Least values were observed in samples with GA as carrier agents. OHC decreased with increase in carrier agents across the study. While experimenting with the freeze-dried powder of pomegranate juice with different carrier agents, Adetoro *et al.* (2020) reported similar values. However, their study reported higher values for oil compared to water which is contradictory to the present study. The difference in values can be attributed to different carrier agents having varied particle structure. Also, there is significant difference among different samples studies for OHC (P<0.05)

Hygroscopicity: Hygroscopicity of powders indicates the ability to adsorb moisture from the surrounding atmosphere.It was reported that lower moisture in spray dried acai powder have tendency to adsorb moisture from surrounding air and become more hygroscopic (Tonon *et al.*, 2008). MD added samples reported highest values for hygroscopicity followed by GAand SPI. Hygroscopicity decreased with increase in carrier agents across the study. Highest value was reported for sample with MD

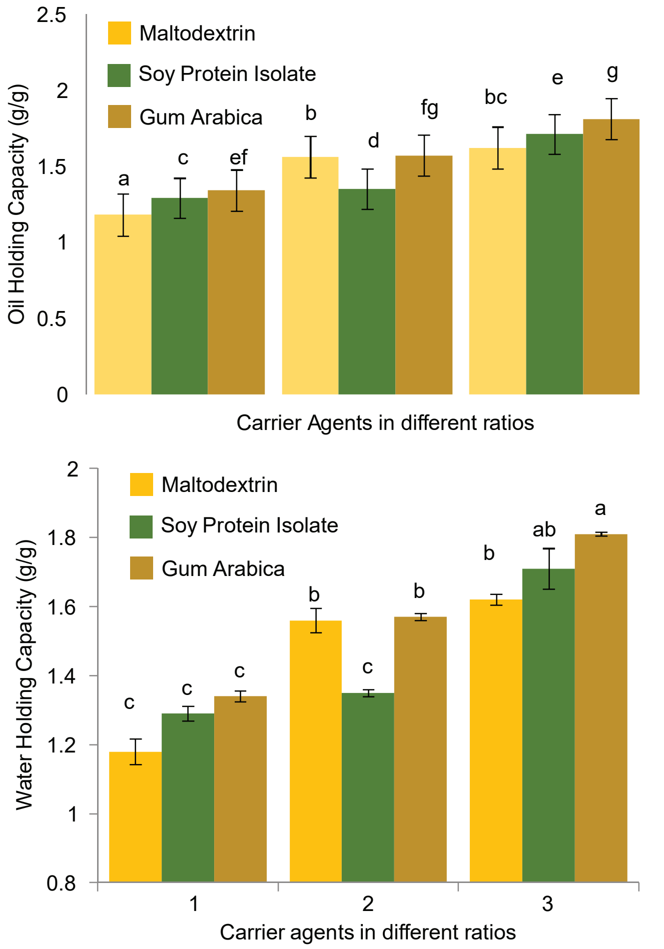

Fig. 4. (a) Oil holding capacity (b) Water holding capacity of ash gourd powder with carrier agents of different ratios. Different alphabets in bar graph apex indicate significant different at *P* <0.05) by Tukey's test)

at 1:10 (18.66) and lowest value for sample added with SPI 1:5 (7.53). Significant difference was reported across all samples by Tukey's test (P<0.05). Caparino *et al.* (2012) reported 18.0 while drying mango pulp using freeze dryer without any carrier agents. In another study by Khalilian Movahhed and Mohebbi (2016) increase in MD in spray dried carrot-celery juice reported reduced hygroscopicity. The study attributed this trend to effect of MD in dried powder as it possesses less hygroscopicity. The present study is in agreement with previous studies reported above.

Color values: Color values are defined by three parameters namely, L*, a* and b* (International Commission of Illumination (CIE, 1986)). Where, L* is a measure of lightness or illuminance which ranges between 0 (black) to 100 (white). a* is one of the two chromatic components range from -120 to 120 (green to red) and b* (blue to yellow). Range of color values reported for ash guard powder with carrier agents in different proportion is shown in Fig. 5.

All powder samples studied with different carrier agents in various proportions showed lightness. Amongst the samples, MD samples were brighter compared to remaining two carrier agents. Also, brightness increased with increase in MD proportion in the sample. Similar trend was observed in samples added with GA but, with lesser brightness values. However, contrary results were observed in case of SPI samples. This might be due to influence of brown color of carrier agent dominating over white color of ash gourd juice. Similar was the case for b* values where its values decreased with increase in SPI. This indicates slightly towards yellow sting for sample color over blue. On the other hand, a* value did not show much significant difference among the carrier agents. Similar values were reported by Lee *et al.* (2017) for spray drying of Asian pear juice powder with different proportion of MD by spray drying.

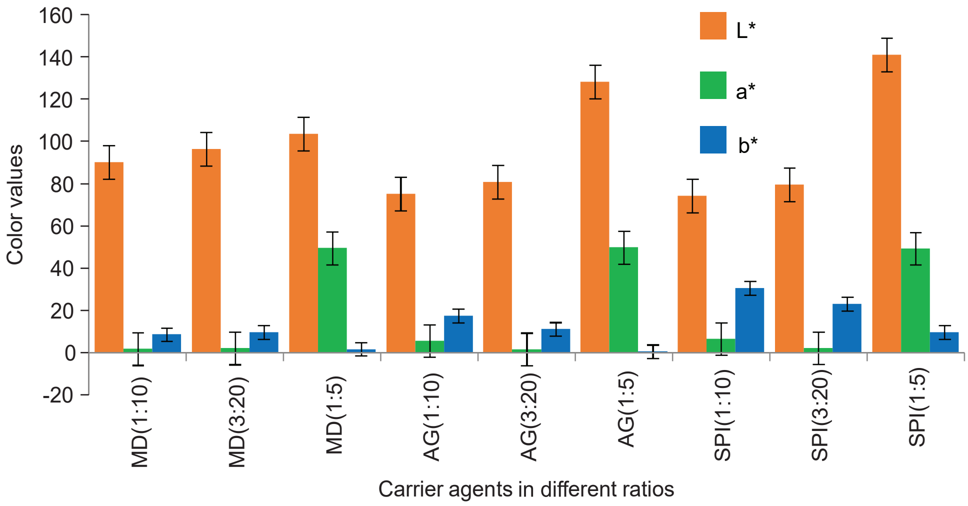

Fig. 5. Color values of ash guard powder with carrier agents of different ratios

Moisture content: Moisture content of dried powder influences physical and chemical property thereby affecting shelf life. Moisture content of dried powder decreased with increase in carrier agents' concentration across the study. SPI as carrier agent reported highest moisture content in resultant AGP. Maltodextrin samples reported least values for moisture content at higher concentration. Trend of decrease in moisture content of AGP with increase in carrier agents can be attributed to water binding capacity of all carrier agents. Lemon by-product aqueous extracts were freeze and spray dried with MD and soy protein by Papoutsis *et al.* (2018). Their study reported that powders from freeze-drying technique had lower moisture content compared to spray drying. Also, MD in combination with soy protein and carrageenan reported lower moisture content compared to MD alone. Through their study, they concluded that encapsulating conditionsand different compositions of the coating agents will have influence on final moisture content of dried powder.

The effect of maltodextrin (MD), soy protein isolate (SPI), and gum Arabic (GA) on the physico-chemical and functional properties of ash gourd powder (AGP) were evaluated in this study. Powder recovery and flowability were highest for GA and MD improved brightness and swelling capacity. The bulk and tapped densities of SPI samples were highest but they were also darker. Moisture content decreased with increasing carrier agent proportions, and MD had the lowest levels of moisture. The water and oil holding capacities differed and MD performed better than GA and SPI. The findings reveal the carrier agents facilitate AGP quality for possible food applications.

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