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Research Article

DRY GRANULATION TECHNIQUE FOR CONVERTING TRIPHALA CHURNA AS GRANULES, TABLETS AND ORGANOLEPTIC EVALUATION

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ABSRACT

The objective of this research work was the conversion of *Triphala churna* into stable, palatable and patient acceptable granules to swallow conveniently by dry granulation methods, using suitable binding agents. The churna granules formulations optimized on the basis of acceptable flow properties of granules. These properties of developed herbal churnas tablets are compared with corresponding marketed product. Developed churna granules were tested for organoleptic evaluation by human volunteers. The churna tablet formulations possess acceptable pharmaceutical properties such as thickness, hardness, friability and weight variation. Stability studies were performed for developed granules as per ICH guidelines and results indicated that the selected formulation were stable.

Keywords: *Triphala churna*, binding agents, granules, organoleptic studies, stability studies.

INTRODUCTION

Churnas are fine powder of Ayurvedic drug or drugs. Among all Ayurvedic dosage forms *churnas* are most prescribed dosage forms. Some of the *churnas* are having large dose, which makes inconvenience to the patients to swallow. Some of the *churnas* stick to the tongue and oral cavity due to inherent adhesive nature. Patients are showing less interest to take herbal *churnas* orally because of their astringent, bitter and pungent taste. *Churnas* being in powder form also suffer stability due to their hygroscopicity. *Triphala churna* is a powder preparation of three myrobalan fruits, (i) *Amalaki* (*Emblica officinalis* Gaertn) (ii) *Haritaki* (*Terminalia chebula* Retz) and (iii) *Bibhitaki* (*Terminalia belerica,* Roxb) in equal proportions[1]. It is widely prescribed as a bowel regulator, purgative, and as an immunity booster[2]. *Triphala* formulation is traditionally prescribed in the form of powder or *churna*, a powder of equal proportions of dried fruits of all the three ingredients mentioned above. Ayurvedic formulary of India specifies the dose of *Triphala* powder to be 3-6 gm per day[3]. But swallowing such large amounts of powder is difficult to the patients.

Bulkiness of herbal formulations shall be reduced by converting the herbal powder into granules by slugging or compaction methods[4]; hence their density can be improved. Dry granulation is energy efficient and suitable for processing pharmaceutical agents that are sensitive to moisture and heat[5]. Slugging technology is to compact the dry powder into large size tablets (called slugs) using heavy duty compression machines under higher force. The slugs so prepared without any wet granulating agents are then size reduced to obtain granules of higher density than original powder.

Taste is an important factor governing the patient compliance. It has gained importance as the most of the drugs are administered through oral route. Administration of unpalatable churnas has hampered by their unpleasant taste particularly in case of paediatric and geriatrics[6]. Unpleasant taste and odour can be masked by using sweetening and flavouring agent.

Hence in the present study, we aimed at conversion of Triphala churna into stable, palatable and patient acceptable granules to swallow conveniently by using dry granulation methods and suitable formulations strategies. Granules will also be formulated with permitted sweeteners, flavours and anti-caking agents and they will be evaluated appropriately including physical stability studies.

MATERIALS AND METHODS

Raw Materials

Dried Myrobalan fruits of *Amalaki* (*Emblica officinalis,* Gaertn), *Haritaki (Terminalia chebula,* Retz) and *Bibhitaki* (*Terminalia belerica* Roxb).

Chemicals

Avicel, PVPK30, Methyl cellulose, Hydroxyl propyl methyl cellulose, starch granules, calcium carbonate, microcrystalline cellulose. Aerosil, croscarmellose sodium, Sodium Saccharin, Propyl and methyl parabens and Vanillin.

Procurement of Herbs

Three herbal ingredients of *Triphala churna* were purchased in the local market, Tumkur and the same were authenticated by Prof. K. Siddappa, Department of Botany, Sree Siddaganga College of Arts, Science and Commerce, Tumkur, Karnataka, India.

Combination of Herbal Ingredients

All the ingredients of *Triphala churna* were subjected for size reduction using the pulveriser. Obtained powders of *Amla, Bibhitaki* and *Haritaki* passed through sieve no. 60.

Preparation of Churna

Formulation of churna was done as per Ayurvedic Formulary of India. Then all the three ingredients are mixed in equal proportion by using planetary mill.

Determination of Physicochemical Properties[7]
Determination of Moisture Content by LOD

Each ingredient (1 gm) was taken in petridish individually and noted the weight (W1). Ingredients were dried in a hot air oven at 100 ºC for 3 hours. Final weight (W2) was noted and the loss in weight is considered as moisture content.

Moisture content was determined using the formula

$$= \left(\frac{W1 - W2}{W1}\right) 100$$

Determination of Total Ash

About 1 g accurately weighed *Triphala churna* was taken in tarred silica dish and incinerated at a temperature not exceeding 450 ºC until free from carbon, then cooled and weighed.

$$\text{Total Ash \%} = \left(\frac{Z-X}{Y}\right) 100$$

Where, X=Weight of empty dish, Y=Weight of *Triphala churna* taken, Z=Weight of empty dish + ash (after completion of incineration).

Determination of Acid Insoluble Ash

To the crucible containing total ash, 25 ml of dilute hydrochloric acid is added. The insoluble matter on an ash less filter paper (Whatman 41) is collected and washed with hot water until the filtrate is neutral. Filter paper containing the insoluble matter is transferred to the original crucible, dried on a hot-plate and ignited to get constant weight in an incinerator. Allowed the residue to cool for 30 minutes and weighed without delay.

$$\text{Acid Insoluble Ash} = \left(\frac{a}{y}\right) 100$$

Where, a= weight of acid insoluble residue, y= weight of *Triphala churna* used.

Determination of Water Soluble Ash

Total ash is boiled for 5 minutes with 25 ml of water; insoluble matter is taken on an ash less filter paper, washed with hot water, and ignited for 15 minutes at a temperature not exceeding 450 ºC. The weight of the insoluble matter is subtracted from the weight of the ash; the difference in weight represents the water soluble ash.

$$\text{Water Soluble Ash} = \left(\frac{a-b}{y}\right) 100$$

Where, a=weight residue after incineration, b=weight of water insoluble residue, y= weight of *Triphala churna* used.

Determination of Alcohol Soluble Extraction

Macerated 5 g of the dried *Triphala churna* with 100 ml of alcohol in a closed flask for twenty-four hours, shaked frequently during six hours and allowed to stand for eighteen hours. Filtered rapidly, taking precautions against loss of solvent, evaporate the filtrate to dryness in a tarred flat bottomed shallow dish, and dried at 105 ºC, to constant weight and weighed. Calculated the percentage of alcohol soluble extraction.

Determination of Water Soluble Extraction

Procedure followed is similar to determination of alcohol soluble extractive, using chloroform-water instead of alcohol.

Determination of Pre-Compression Parameters of Ingredients
Determination of Tapped Bulk Density

Each ingredient (10 gm) was taken in a measuring cylinder and the volume before and after tapping 100 times was noted. Tapped bulk density was calculated based on the following formula.

Tapped bulk density = weight of the ingredient / Tapped volume

Determination of Angle of repose
Approximately 10 gm of each ingredient was taken and passed through the funnel to obtain a pile of the powder. The height (h) and radius (r) of the pile of the powder were noted down. The angle of repose (θ) was calculated using the formula

$$(\theta) = \text{Tan}^{-1} (h/r)$$

Formulation of Three Herbal Churna Granules

Formulation of churnas was done as per Ayurvedic Formulary of India. In addition sweetener, flavouring agent and disintegrating agent, binding agent, lubricants, preservatives and cooling agent were added.

Method of Preparation of Churna Granules & Tablet

Churna granules & tablets were prepared as follows. The herbal powders and all other ingredients were sifted through sieve # 60. Each ingredient with lubricants is weighed and mixed uniformly using the planetary mixer running for 20 minutes. The mixture is subjected for slugging for converting the powder mixture into slugs. The obtained slugs were passed through the dry granulator to get the granules. Finally tablets were compressed on a 10 station mini rotary tableting machine (Shakti Pharma Tech Pvt. Ltd, Ahmadabad) with flat-shaped punches.

Evaluation of Pre-Compression Parameters of Granules

Bulk density (Db)
It is the ratio of weight of powder to bulk volume. The bulk density depends on particle size distribution, shape and cohesiveness of particles. Accurately weighed quantity of powder was carefully poured into graduated measuring cylinder through large funnel and volume was measured which is called initial bulk volume. Bulk density is expressed in gm/cc and is given by,

$$Db = M / Vo$$

Where, Db= Bulk density (gm/cc), M = the mass of powder (g), Vo= bulk volume of powder (cc).

Organoleptic evaluation of herbal churna and granules using human volunteers

Organoleptic evaluation studies were carried out after obtaining permission from Institutional Ethics Committee for Human Research, Ashwini Ayurvedic Medical College and Research Centre, Tumkur, Karnataka, India. Volunteers were given *Triphala churna* powder and asked to keep in the oral cavity to evaluate the taste and acceptability. Volunteer rinsed the oral cavity with

drinking water and a gap of 30 minutes is given to evaluate developed *Triphala churna* granule.

Evaluation of post compression parameters[8]

Thickness

Control of physical dimension of the tablet such as thickness and diameter is essential for consumer acceptance and tablet uniformity. The thickness of the tablet was measured using Vernier callipers. It is measured in mm.

Hardness

The Monsanto hardness tester was used to determine the tablet hardness. The tablet was held between a fixed and moving jaw. Scale was adjusted to zero; load was gradually increased until the tablet fractured. The value of the load at that point gives a measure of hardness of the tablet. Hardness was expressed in kg/cm^2.

Friability (F)

Tablet strength was tested by Roche friabilator. Pre weighed tablets were allowed for 100 revolutions (4 min), taken out and were dedusted. The percentage weight loss was calculated by rewriting the tablets. The percentage of friability was then calculated by,

$$F=\left(\frac{Wint-Wfina}{Wint}\right)100$$

Where, F = Percentage friability, W_{init} = Initial weight before friability test, W_{fin} = Final weight after friability test.

Stability Studies

Stability studies of pharmaceutical products were done as per ICH guide lines. These studies are designed to increase the rate of chemical or physical degradation of the drug substance or product by using exaggerated storage conditions.

Selected formulations were placed in a met pet laminates and sealed using sealing machine, stored at different storage conditions at elevated temperatures such as 40 ºC ± 2 ºC / 75% ± 5% RH for 90 days. The samples were withdrawn at intervals of fifteen days and checked for physical changes.

Table 1: Properties of individual ingredients of *Triphala churna*

Ingredients	Moisture Content (%)	Tapped Bulk Density (gm/cc)	Angle of Repose
Amla	0.06	0.66	$37^0 95''$
Bibhitaki	0.08	0.55	$39^0 35''$
Haritaki	0.06	0.63	$41^0 98''$

Table 2: Physicochemical properties of *Triphala churna* without excipients

Properties	Value (%)
Total ash	14.41
Water soluble ash	02.70
Acid insoluble ash	03.20
Water soluble extract	36.14
Alcohol soluble extract	19.40

Table 3: Composition of Triphala *churna* granules by dry granulation technique to improve patient compliance

Formulation Code	T1	T2	T3	T4	T5	T6	T7
Triphala churna (gm)	250	250	250	250	250	250	250
Binding agent (gm)	25 (Avicel)	25 (PVPK30)	25 (MC)	25 (HPMC)	25 (Starch granules)	25 ($CaCO_3$)	50 (MCC)
Croscarmellose sodium (gm)	5	5	5	5	5	5	5
Saccharin sodium (gm)	25	25	25	25	25	25	25
Vanillin (gm)	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Propyl paraben (gm)	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Metyl paraben (gm)	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Magnesium stearate (gm)	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Talc (gm)	12.5	12.5	12.5	12.5	12.5	12.5	12.5
Mannitol (gm)	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Aerosil (gm)	5	5	5	5	5	5	5

Table 4: Properties of *Triphala churna* formulations before (bulk) and after converting into granules by slugging method

Formulation code	Sample	Moisture content (%)	Angle of repose	Tapped Bulk density (gm/cc)	Carr's index (%)
T1	Bulk	--	44º00″	0.573	27.43
	Granules	0.10	32º12″	0.689	12.04
T2	Bulk	--	44º58″	0.625	27.30
	Granules	0.04	34º05″	0.800	10.70
T3	Bulk	--	45°76″	0.509	30.40
	Granules	0.06	29°03″	0.571	07.20
T4	Bulk	--	49°88″	0.645	32.71
	Granules	0.04	36°57″	0.869	20.70
T5	Bulk	--	48°16″	0.645	26.20
	Granules	0.04	34°76″	0.689	19.40
T6	Bulk	--	38°65″	0.588	38.26
	Granules	0.08	23°74″	0.606	08.41
T7	Bulk	--	43°13″	0.526	30.98
	Granules	0.10	37°37″	0.625	11.20

Table 5: Percentage yield of *Triphala churna* granules obtained from its slugs using dry granulator

Formulation code	Percentage of granules (%)	Percentage of fines (%)
T1	68.93	29.77
T2	76.54	23.30
T3	68.41	30.41
T4	64.01	34.99
T5	72.02	28.05
T6	68.61	30.61
T7	64.10	33.95

Table 6: Paired sample test report for comparison of developed *Triphala churna* granules (T3) and marketed product (MP)

		Mean rating	t	df	Sig.
Pair 1	Appearance of T3 Appearance of MP	3.95 3.43	-0.305	40	0.004
Pair 2	Taste of T3 Taste of MP	4.19 2.68	-11.954	40	0.001
Pair 3	Smell of T3 Smell of MP	4.53 3.91	0.518	40	0.005
Pair 4	Overall Acceptability of T3 Overall Acceptability of MP	4.29 3.46	-5.776	40	0.001

Table 7: Comparison of physical properties of developed *Triphala churna* tablet formulations (T-1 to 7) and marketed product

Formulation Code	Marketed product	T1	T2	T3	T4	T5	T6	T7
Thickness (mm)	4.5±0.02	4.6±0.03	4.7±0.01	4.7±0.02	4.5±0.02	4.6±0.03	4.7±0.02	4.6±0.03
Hardness (kg/cm^2)	4.7±0.3	6.4±0.2	6.3±0.25	6.4±0.2	6.1±0.25	5.9±0.34	6.0±0.3	6.4±0.2
Friability (%)	0.4	0.30	0.25	0.41	0.41	0.39	0.42	0.41
Weight variation (mg)	670±20	682±22	678±19	686±15	675±19	672±20	680±12	672±21
Disintegration time (min)	20.0	12.0	13.5	11.9	14.0	12.5	13.5	12.8

Figure 1: *Triphala churna* powder, slugs and granules (left to right)

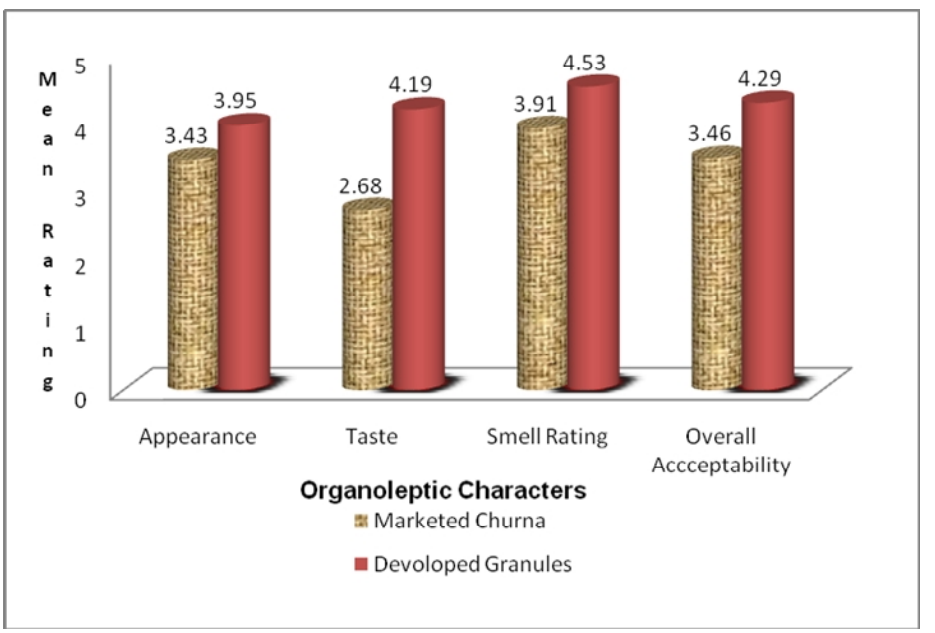

Figure 2: Comparison of organoleptic characters of developed *Triphala churna* granules (T3) and marketed *Triphala churna*

RESULTS AND DISCUSSION

Each individual ingredient of *Triphala churna* is subjected to determination of moisture content, tapped bulk density and angle of repose and their values are shown in Table 1. Angle of repose values ranged from $37^0 95$" to $41^0 98$" for ingredients this indicates the ingredients possess fair to possible flow properties inherently. *Triphala churna* is prepared by mixing equal quantities of herbal powders of *Amla*, *Bibhitaki* and *Haritaki*. Total ash value, water soluble ash, acid insoluble ash, water soluble extract and alcohol soluble extract of *Triphala churna* are determined and their values are mentioned in Table 2.

Triphala churna is converted into its granules by using seven binding agents such as Avicel, PVPK30, methyl cellulose (MC), Hydroxyl propyl methyl cellulose (HPMC), starch granules, calcium carbonate and microcrystalline cellulose (MCC). Starch granules are prepared in our laboratory by passing the wet mass of starch through sieve no. 14. Croscarmellose sodium is used as disintegrating agent. Saccharin sodium and Vanillin are used as sweetening and flavouring agents, respectively. Propyl and methyl parabens are added for the purpose of preservation. Magnesium stearate and talc are served as lubricants, mannitol is a cooling agent. Aerosil functions as anti-caking agent to stabilize the formulation. Formulations of *Triphala churna* are mentioned in the Table 3.

Triphala churna formulation ingredients are mixed by planetary mixture, the obtained blend is subjected for slugging to get slugs by 10 station rotary compression machine. Initially 5 % binding agent (based on quantity of Triphala *churna*) was tried. Formulations containing 5 % of binding agent could not able to provide slugs with sufficient hardness where as formulations containing 10 % of binding agent produced slugs of required hardness. However, in case of formulation T7 where MCC used as binding agent, higher quantity of binding agent was required to get slugs with sufficient hardness, i.e. 10 % of MCC could not produce slugs whereas formulation containing 20 % of MCC were able to produce the slugs. The obtained slugs are then placed in a dry granulator (Cronimach Machinery) to get the granules of *Triphala churna* formulations (Figure 1).

Properties of different blends of *Triphala churna* formulations (bulk) and their granules obtained after slugging were determined and the results are mentioned in the Table 4. A perusal of Table 4 indicates angle of repose of granules was reduced compared to their corresponding bulk in all the formulations (T1 to T7). For example angle of repose of bulk of formulation T1 was 44º00″ after converting into granules it was reduced to 32º12″. This indicates the flow properties of powder blend had been improved by converting into granules. While similar results are observed comparing compressibility index values for bulk and granules i.e. compressibility index values of formulations (T1 to T7) were decreased for granules while comparison with bulk. Compressibility index values of bulk of T1 formulation was 27.43% after converting into granules it was reduced to 12.04%. Decrease in compressibility index values indicates the good flow of granules prepared. Among seven binding agents used in formulation, based on angle of repose and compressibility index values formulation containing methyl cellulose (T3), starch granules (T5) and calcium carbonate (T6) produced good flowable granules comparatively. Starch granules are used as binding agent instead of starch powder with the intention that coarse granules provide more efficient binding site than the powder. The observations showed that sufficient harder granules of churna formulations are obtained with the starch granules compared to starch powder as binding agent.

To know the efficiency of binding agent selected and suitability of dry granulator used, the percentage of granules obtained from the slugs was calculated. Percentage of granules was calculated by the following formula

Percentage of granules = (weight of the granules obtained / weight of the slugs taken) × 100

As shown in the Table 5, the percentage of granules obtained was in the range of 64 to 76 % for seven Triphala churna granule formulations which indicates suitability of the use of binding agents and slugging as a choice of method for preparation of granules. In efficient binding agent may fail to produce hard enough slugs and dry granulator may convert such slugs into less percent granule and more percent fines.

Addition of sweetener and flavouring agent is a simplest way to mask the taste of the churnas this method was selected in the study. After taste evaluation studies, volunteers reported that Triphala churna as such was not acceptable and pungent in taste. But the developed Triphala churna granule formulation (T3) was of acceptable with suitable taste. This is achieved by addition of 10 % saccharin sodium and 1 % vanillin as flavouring agent. Additionally, the conversion of granules reduced the effective surface area of the churna having pungent taste that come in contact with the tongue upon oral intake. All the volunteers concurrently accepted the taste of developed Triphala churna granule formulation.

Volunteer were provided sensory evaluation forms to mention their opinion of organoleptic properties of developed Triphala churna granules. In case of organoleptic evaluation, volunteers asked to rate from 1 to 5 (high rating value means high acceptability of organoleptic property). Feedback from the volunteers (n=41) was collected and subjected to statistical analysis using the SPSS statistical version 16.0 (SPSS) In., Chicago IL and results are shown in Table 6 and Figure 2. As per statistical analysis the developed Triphala churna granules possess better appearance (mean 3.95) than marketed Triphala churna powder (mean 3.43) ($p<0.01$). Similarly, taste and smell of Triphala churna granules are better than Triphala churna powder. On statistical comparison, Triphala churna granules (Formulation T3) are overall accepted than marketed product with statistical significance ($p<0.001$).

All the developed Triphala churna granule formulations were converted into tablets using 10 station compression machine. The properties of developed *Triphala churna* tablets such as Hardness, Friability and Weight variation similar to the marketed product (ZANDU). However, disintegration time of developed tablets was less ranging from 11.9 to 14 min, as compared to disintegration time of marketed product i.e. 20 min (Table 7). Among seven formulations T3 & T6 posses better flow properties, hence these two formulations are selected for stability studies. Even after 90 days, and there is no change in the physical appearance and properties of the developed *Triphala churna* granules.

CONCLUSION

The present study confirms the feasibility of use slugging method to convert *Triphala churna* into granules & Tablet dosage form. Saccharin sodium 10 %, vanillin 1 % could able to improve the organoleptic properties of the *Triphala churna* granules. Among the binding agents used calcium carbonate 10 % and methylcellulose 10 % were produced a granules with suitable hardness and good flow properties. Therefore, suitable formulation strategy can overcome the unacceptability of *Triphala churna* by consumers.

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