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OPTIMIZATION OF MOISTURE CONTENT TO STRENGTHENING THE GRANULES PREPARED IN ROTARY DRUM GRANULATOR

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ABSTRACT

In wet granulation process, granules are prepared in rotary drum granulator by adding a liquid binding agent as an aqueous solution of corn starch. Various factors, which affecting the production and strength of granules are particle size, moisture (liquid binder), residence time (time of rotation) and binder. Liquid binding agent plays a significant role in determining the strength of granules. To get better production and strength of granules several experiments are carried out with varying moisture content by keeping constant time of rotation and feed quantity. Increase in moisture leads to increase in production and strength of granules until an optimum condition is reached where nucleation and layering is done perfectly. Further increase in moisture leads to less strength and more plasticity this is due to over coalescence of granules. To determine the strength of granules a very important test i. e. Drop test is done with repeated droppings and with varying height of dropping for different size of granules at wet and dried conditions. From the experiments and drop tests on different size of granules at different conditions, an optimum condition of moisture content for feed is obtained, which is responsible for more production and added strength of granules.

Key words: Strength of granules, Moisture, Time of rotation, Nucleation, Layering, Coalescence, Rotary drum granulator, Drop test.

INTRODUCTION

Granular materials are also used in many industries extensively, such as pharmaceutical engineering, polymer, and chemical engineering. Rotating drums are used in chemical, pharmaceutical, and mechanical industrial engineering for processing granular materials, such as for mixing, granulation, segregation, coating and drying.

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Granules, which are referred in this paper done by wet granulation using an aqueous solution of corn starch as liquid binding agent to zirconium oxide powder and carbon are prepared in rotary drum granulator. Usually granules[1] provide more surfacial area[2], porosity[3] when compared to briquettes as feed for gas solid reaction, which are mostly using in chemical industries. Granulation is a method to convert fine particles into physically stronger and larger agglomerates[4] called as granules[5] with improved appearances, good flow property, better compression characteristics, mixing uniformity and reduce dustiness[6,7].

To perform particle size enlargement, a number of primary particles are to be bound to form agglomerate. This can be achieved using cohesive forces of the powder only (dry granulation)[8] or using capillary and viscous forces by addition of a binder liquid (wet granulation)[9].

Dry granulation

Dry granulation is a technique chosen, for the powder which is sensitive to moisture. In dry granulation powder particles have to come in close contact with each other, to have the Vander Waals forces create the agglomerate. Slugging, which means the compression of a powder, is mostly used to obtain bonding. This can be performed in a roller compacter, in which powder particles are fed between two large rolls and thereby compacted. Sometimes even the presence of adsorbed layers of moisture is enough to bind particles.

Wet granulation

In this process, powder particles are mixed with liquid binders to produce granules. These granules are formed due the formation inter-particle bonds between them. Liquid binder should be distributed through the powder by the help of mechanical agitation created by the granulator. A thorough mixing of binders and particles will develop liquid bridges among particles. As more liquid is added the tensile strength of these bonds will increase. These surface tension forces and capillary pressure among liquid particles (liquid bridges) are primarily responsible for initial granule formation and strength. In wet granulation, the amount of binder liquid used is usually defined as the ratio of binder liquid mass to the powder mass. However, prediction of the appropriate amount of water to obtain the desired granule size is difficult, due to the fact that this amount is affected by many variables. Examples of these variables are powder mixture properties, such as moisture content and particle size, and liquid characteristics, like viscosity.

Granulation mechanism

Granules are formed due to formation of bonds between powder particles and these

bonds must be strong enough to prevent breakdown of the final dried granules to powder in subsequent handling operations. There are five key mechanisms operating during the agglomeration process, but generally more than one will be applicable to any particular system as shown in Fig. 1[10]. They are elaborated as follows:

Adhesion and cohesion forces

These forces help in formation of immobile liquid films that act like binder bridges.

Liquid bridges

This is particle adhesion forces that arise from surface tension of capillary pressure and the liquid/air system and also attraction between the surface of the solid substance and water.

Solid bridges

Solid bridges are like the bonds that are formed by the hardening of binder under high temperature. The force of the cohesion depends on a diameter of the contact area and strength of the bridge material.

Fig. 1: Process principle of granulation

Van der waals forces

Attractive forces between solid particle surfaces. Decrease in particle size stables agglomerates.

Form-closed bonds or interlocking bonds

Such as fiber and bulky particles gives interlocking bonds.

Theory of granule formation and growth

In order to judiciously select and optimize any pelletization/granulation process, it is important to understand the fundamental mechanisms of granule formation and growth. Different theories have been postulated related to the mechanism of formation and growth of granules. Some of these theories are derived from experimental results while others are confined to visual observations. Results obtained from the experiments with some form of tracer technique are regarded as acceptable and convincing. As the conventional granulation, the most thoroughly studied, most classified pelletization process, which involves a rotating drum, a pan or a disc, has been divided into three consecutive regions: nucleation, transition and ball growth.

However, based on the mechanism of granule formation and growth, the following steps were proposed:

Nucleation[11-13]

Whenever a powder is wetted with liquid nucleation begin, which is a common stage of pelletization/granulation process. The primary particles are drawn together to form three-phase air-water-liquid nuclei and are attached together by liquid bridges, which are pendular in nature. The bonding strength is improved by reduction of particle size. The size of the primary particles, the moisture content, the viscosity of the binding particles, the wettability of the substrate and the processing conditions, such as tumbling and drying rates, influence the size, the rate and the extent of nuclear formation. Both the mass and the number of nuclei in the system change as a function of time, which is an important feature of nucleation. Nucleation is followed by a transition phase and the growth mechanism affecting the transition region is coalescence and layering as shown in Fig. 2A.

Coalescence[14]

It is defined as the formation of large-sized particles by random collision of well-formed nuclei, and the mechanism requires slight excess moisture on the nuclear surface.

Although the number of nuclei is progressively reduced, the total mass of the system remains unchanged during this step, Fig. 2B.

Layering[15-16]

It is a slow growth mechanism and involves the successive addition of fragments and fines on an already formed nucleus. In the layering step, the number of particles remains the same, but the total mass in the system increases due to increasing particle size as a function of time. The fragments or fine particles can be formed by particle size reduction that occurs due to attrition, breakage and shatter. The fines and the fragments that are produced through size reduction are picked up by large pellets. Production of fines and subsequent coalescence and layering continues until the number of favourable collisions declines rapidly, thereby leading to a reduction in the rate of growth of the pellets. At this point the third phase, the ball growth region, is reached. In the ball growth phase the main mechanism affecting the slow growth of agglomeration is the abrasion transfer, Fig. 2C.

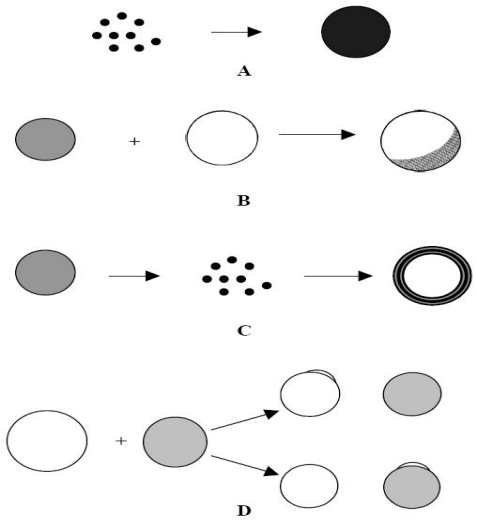

Fig. 2: Granule growth mechanisms: (A) Nucleation, (B) coalescence, (C) layering and (D) Abrasion transfer

Abrasion transfer[17]

Abrasion transfer involves the transfer of materials from one granule formed to another without any preference in either direction. This situation does not result in a change in the total number or mass of the particles. The particles, however, undergo a continuous change in size as long as the conditions that lead to the transfer of material exist, Fig. 2D.

Granulation equipment

Granulation and agglomeration are the terms generally used to describe the process of particle formation or size enlargement. There are typically four main types of wet agitated granulating equipment based on the agitation process, they are drum granulators[18], pan granulators[19], fluidized bed granulators[20] and mixed granulators (or high shear granulators)[21].

Rotary drum granulator

Rotation drum granulators are mainly used for large capacities and heavy-duty materials where the binders are sprayed continuously over the rolled product during processing. The equipment is inclined at an angle up to 10° to help emptying the granules formed. The speed of rotation n is (0.30-0.55) n_c i.e. critical speed, generally n is 8 to 20 rpm. The degree of filling is low (2-3%) and the length to diameter ratio (L/D) of drums is 2-5. The usage of maximum drum capacities can be noticed in chemical and metallurgy industries where drums with diameters up to 3m and length of 15 m are used to attain a capacity of 100 tons/h, capacity of the rotating drum depends on the properties[22] of the product and agglomerates processed. The key parameters that affect drum granulation process are moisture content, initial size of feed particles, rotational speed, residence time, and binder viscosity[23].

Moisture content

The moisture content should be at optimum levels depending on the material. Apparently, there is an optimum amount of liquid binder to produce granules. To a certain extent, the more liquid used the denser the granules will be, leading to increased sphericity. Therefore, the amount of binder not only determines granule size, but also granule shape and dry granule strength.

Rotational speed

The rotational speed should be at optimum levels depending on the material. The optimal speed will allow particles to move in a cascading motion which increases the chance of coalescence and agglomeration.

Residence time

The residence time is proportional to particle growth, increasing residence time will ultimately increase growth and vice versa.

Binder viscosity

Binder liquid viscosity determines the granulation process to a significant extent. A high viscosity affects the consolidation of the granule. The higher viscosity, the less compacted the granule will be, due to the decreased mobility of the binder fluid [24,25]. The reduced mobility also leads to decreased binder distribution, leading to a broad granule size distribution. Another effect of a highly viscous binder liquid is the increased wet granule strength this can result in a less deformable material, resulting in a less spherical shape.

Binder viscosity also influences granulation i.e. more viscous binders cause granulation to occur to a higher degree. The size enlargement process occurring in rotating drum is described as follows agglomeration process occurs on bed of a rotating drum. Initially seed material is feed onto the drum to which binding agent is later sprayed through a spraying system. Due to continuous rotation the material becomes uniformly moist and when further material is sprayed on to the bed, agglomerates are formed. Heat in the system can be controlled by the help of air fans as shown in Fig. 3. After achieving the desired size, the granules are deposited on a vibratory screen separator where the granules are segregated into product and undersize granules. The product size granules are taken out of the processing area to storage. The undersize particles are directed to the granulating drum for further size enlargement.

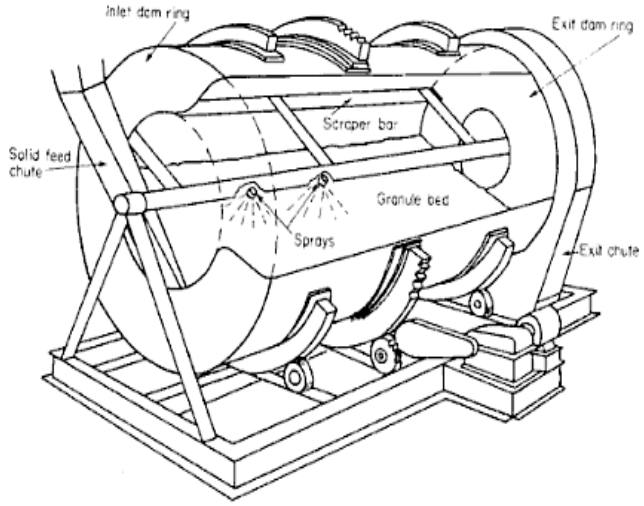

Fig. 3: Drum granulator

The advantages and disadvantages of rotating drum granulator are given as follows:

Advantages of rotating drum granulator are –

(i) Good construction and versatility

(ii) Large capacity and good control

(iii) Continuous or batch processing

Disadvantages of rotating drum granulator are –

(iv) Relatively high energy required

(v) Large dimensions (space) and inefficient use of total volume

(vi) Non-uniformity of agglomerate size

Quantifying pellet strength and durability

In the investigation of the characterization of a quality pellet, it was evident that a number of definitions exist regarding the strength and durability of pellets. Kaliyan and Morey[26] have presented clear definitions of each.

Strength

Strength refers to both the compressive and impact resistance of a pellet.

(A) Compressive resistance: Compressive resistance testing simulates the loading due to self-weight in storage and the crushing of pellets in a screw conveyor. Compressive resistance, also referred to as hardness, is defined as the maximum compressive load that a pellet can incur before cracking. Compressive resistance is modeled using a diametrical compression test in which a single pellet is placed between two flat, parallel platens and an increasing load is applied at a constant rate until fracture. The load at fracture is read off of a recorded stress-strain curve and referred to as the compressive strength of the pellet.

(B) Impact resistance: Impact resistance testing models the impact forces induced on pellets during handling in the filling of silos, bins or storage bays when pellets are dropped either on a hard floor or onto one another.

Several methods have been used to establish the impact resistance of densified materials. All involve dropping a single particle several times from an established height and recording the mass or number of pieces retained above a specified particle size. ASTM

method D440-86 of a drop-shatter test for coal was employed[27] for testing the durability of biomass logs. An impact resistance index (IRI)[28] was then calculated using an equation (1).

$$IRI = 100\ (N/n) \quad \ldots(1)$$

Where N = Number of drops

n = Total number of pieces after N drops

Since the standard number of drops employed by Li and Liu[27] was always two, the maximum value of IRI was 200. Pellets were dropped from a height of 1.85 m onto a metal plate four times. Impact resistance was defined as the percentage of the initial weight retained after dropping.

Durability

Durability is defined as the abrasive resistance of a densified product. Durability, as defined herein, is the most prevalent form of pellet quality analysis employed by pellet manufacturers and is used to adjust parameters during the pelleting process.

Hardness test

It is known that hardness reflects the resistance of material to its permanent deformation. Hardness of agricultural processed materials is measured based on crushing test. Kaliyan and Morey investigated major factors that contribute to strength and durability of densified product, and they unveiled four major parameters: compression time, particle size distribution, moisture content, and compaction conditions.

A machine that is generally used for measuring the mechanical strength of materials (such as compressive strength and tensile strength) can also be used for determining the hardness of pellets. During the hardness test on pellets, the maximum load to break a pellet will be recorded. The Meyer hardness (MPa) is defined as the applied force (N, when the pellet is crushed) divided by the projected indentation area, knowing the indentation depth and the initial diameter of a pellet's cross section[29]. The maximum breaking force and the Meyer hardness of the pellets can be obtained from a typical force-displacement graph displayed during the test.

Drop test – Theoretical development

Modes of pellet or granule breakage

Two major types of breakage are considered in pellets, volume breakage and surface breakage.

Volume breakage

In volume breakage the pellets are broken into smaller pieces including a fine dust. This happens along the cracks, line of weakness when pellets experience impact force. After breakage the new fragments have smaller mean length than the pellet. Dural[30,31] is an example of a device that imparts severe impact and shear on pellets to cause volume breakage.

Surface breakage

For the case where the impact force is not large enough surface breakage happens. In this case only abrasive forces cause chipping and removal of dust and fines from the surface of the pellets. For instance, vibration may cause surface breakage. The degree of dust generation in Surface breakage is less than in volume breakage. Tumbler[32] is a good example of surface breakage. Pellets tested in tumbler remain in their original shape. Only small amount of dust is produced in Tumbler due to surface breakage. From our observation these dusts and fines are mainly from the surface or corners of pellets.

In a drop test both kinds of breakages, volume and surface breakages happen depending on the impact force, which is affected by the mass of pellets and height in which pellets are dropped from.

EXPERIMENTAL

Drop tests with repeated droppings

A few samples of pellets are taken and done repeated droppings until the surface and volume breakage of pellet is done and noted number of drops.

Drop tests with varying drop height

Few samples of pellets are taken and done repeated droppings with varying drop height from 45 cm to 180 cm and noted number of drops.

Drop tests with varying sample mass or size

Again few samples of pellets are taken and it is done repeated droppings with varying sample size 3 mm and 6-10 mm with varying drop height 45 cm and 180 cm and noted number of drops.

Experimental procedure

Feed: A Feed composition of ZnO_2, carbon, starch, moisture is thoroughly mixed in pug mill.

Experimental Feed quantity : 5 Kg

Feed is divided into two halves (2.5 Kg + 2.5 Kg) are added into two successive and different time intervals depending on type of experiment.

Moisture added

Varying moisture content in two time intervals 5 and 10 minutes is added, respectively.

Time of rotation

Time of rotation is fixed 5 and 10 minutes, respectively for two time intervals.

Experimental rotary drum granulator

Rotary drum granulator having length 90 cm and diameter of 36 cm, its L/D Ratio is 2.5. It is rotating in anti-clockwise direction placed in between two rollers rotating clock wise direction with the help of 2HP Motor as shown in Fig. 4. It rotates 31-33 per minute.

Fig. 4: Samples of different size of granules before and after coking

Coking

Natural dried granules are loaded into a retort. The top lid of retort is bolted and nitrogen inlet and exhaust lines are assembled, the retort is now placed in a furnace. The nitrogen metering pipe is connected and the system is leak tested by pressurizing to 2 psig, a cooling water line is also connected. The system is flushed with nitrogen for ½ hr. the charge is then soaked at 650^{o}C for 12 hrs to remove off gases N_2, H_2O and CO_2. The furnace is

switched off and granules are allowed to cool in the furnace for 6 hrs. It is then cooled for 36 hrs in a cooling station. This process decomposes and removes starch in the form of off gases (CO_2, H_2O) as per the following reaction. Porous granules are obtained with this process as shown in Figs. 5-6.

$$(C_6H_{10}O_5)n \longrightarrow 6C + 5H_2O$$

Fig. 5: Samples of 6-10 mm granules before and after coking

Fig. 6: Samples of 3 mm granules before and after coking and sample of different size of granules 3 mm and 6-10 mm

Set of experiments with varying moisture

MA (50 + 150)

Total feed quantity : 5 Kg

Total moisture added : 200 mL

Total time of rotations : 15 min

Procedure

Take half quantity of feed (2.5 Kg) and sprinkle 50 mL of moisture for MA (50 + 150) i.e., (2% of feed), 100 mL of moisture for MA (100 + 100) i.e., (4% of feed) and 150 mL of moisture for MA (150 + 50) i.e., (6% of feed) on to the feed in Rotary drum granulator and make it to rotate for a time interval of 5 min. Then add the another half quantity of feed (2.5 Kg) along with 150 mL moisture for MA (50 + 150), 100 mL moisture for MA (100 + 100) and 50 mL moisture for MA (150 + 50), on to the feed and make it to rotate for a time interval of 10 minutes. Remove the feed from Rotary drum granulator and put into sieve analysis to obtain different size granules (3 mm and 6-10 mm). Weigh and note down the weight of respective (3 mm and 6-10 mm) wet granules as shown in Table 1.

Table 1: Weight of different size of wet granules with varying moisture content

S. No.	Experiment type	Weight of wet granules (3 mm)	Weight of wet granules (6-10 mm)
1	MA (50 + 150)	2	0.2
2	MA (100 + 100)	2.1	0.9
3	MA (150 + 50)	2.3	2.2

Table 2: Drop tests on varying moisture

S. No.	Experiment type	Number of drops (3 mm)		Number of drops (6-10 mm)	
		45 cm	180 cm	45 cm	180 cm
1	MA (50 + 150)	130, 186, 263	11, 8, 12	28, 16, 20	4, 3, 4
2	MA (100 + 100)	210, 184, 167	27, 30, 24	61, 54, 48	6, 7, 5
3	MA (150 + 50)	430, 378, 514	31, 22, 33	196, 140, 168	7, 6, 7

RESULTS AND DISCUSSION

From Table 1 and Fig. 7 shows production of different size of granules at different moisture conditions. In that more production of two different sizes of granules is achieved at MA (150 + 50) condition.

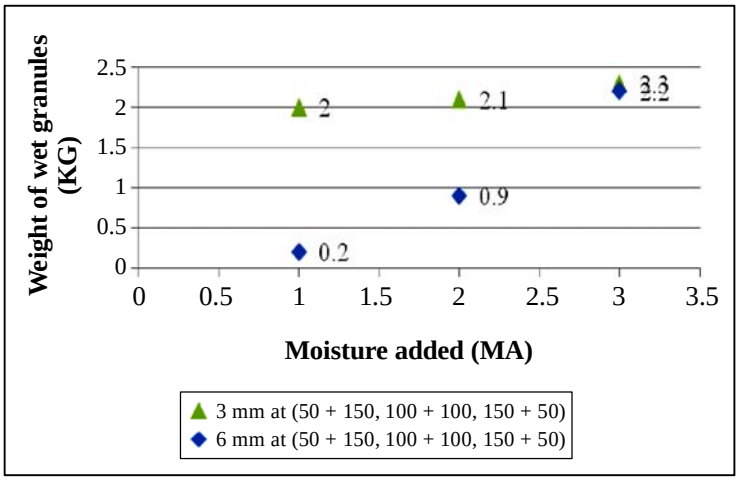

Fig. 7: Weight of different size of wet granules (3 mm & 6-10) mm vs moisture added (MA)

Number of droppings of different size of wet granules with varying moisture added (MA) from different heights 45 cm and 180 cm is done as shown in Table 2.

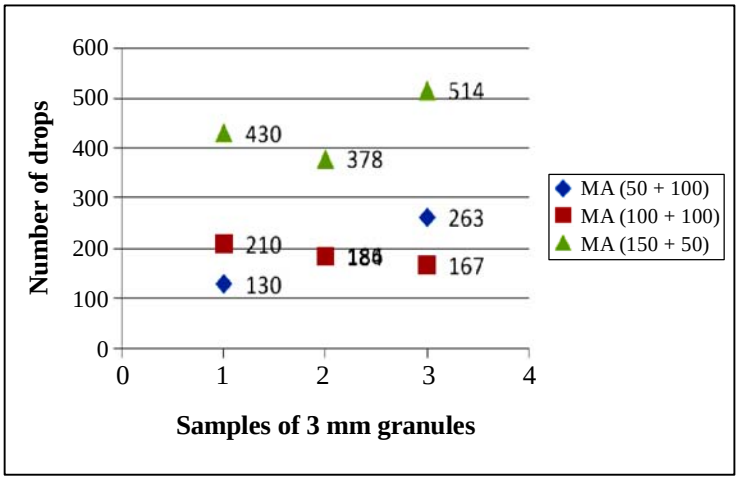

Fig. 8: Number of drops from 45 cm vs samples of 3 mm wet granules

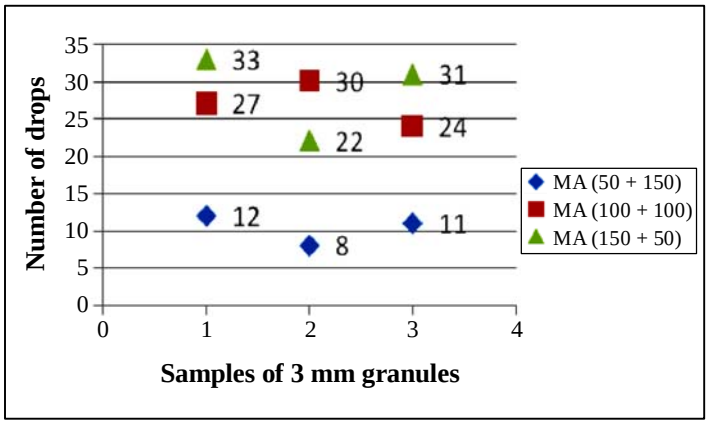

Fig. 9: Number of drops from 180 cm vs samples of 3 mm wet granules

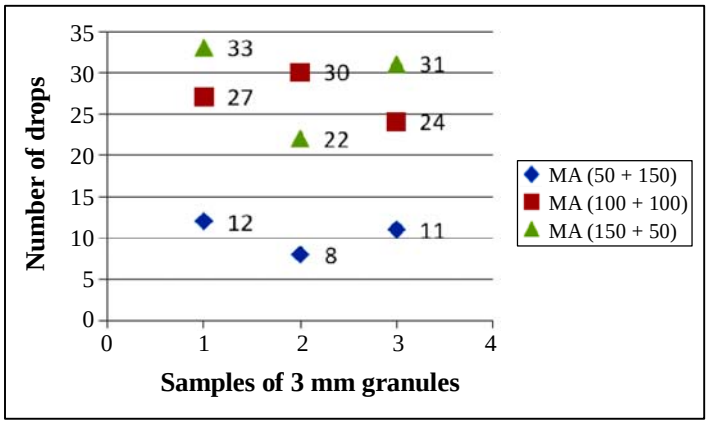

Fig. 10: Number of drops from 45 cm vs samples of wet granules (6-10 mm)

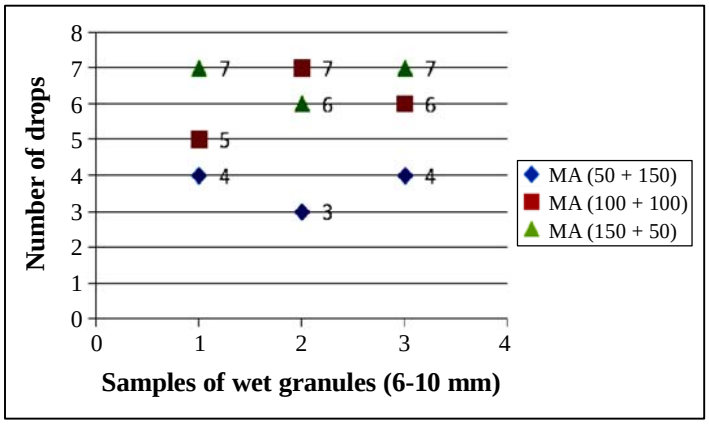

Fig. 11: Number of drops from 180 cm vs samples of 6-10 mm granules

Set of experiments with varying moisture content after coking

From Fig. 11 and Table 3, we can observe decrease in weight of granules after coking when compared to before coking due to loss of moisture and starch in coking.

Table 3: Weight of different size of dried granules with varying moisture after coking

S. No.	Experiment type	Weight of dried granules (3 mm)	Weight of dried granules (6-10 mm)
1.	MA (50 + 150)	1.9	0.1
2.	MA (100 + 100)	2.0	0.7
3.	MA (150 + 50)	2.1	2

Table 4: Drop tests on different size of dried granules with varying moisture after coking

S. No.	Experiment type	Number of drops (3 mm)		Number of drops (6-10 mm)	
		45 cm	180 cm	45 cm	180 cm
1.	MA (50 + 150)	7, 9, 5	2, 1, 2	3, 3, 5	1, 1, 1
2.	MA (100 + 100)	10, 11, 17	2, 2, 1	5, 6, 4	1, 2, 1
3.	MA (150 + 50)	15, 21, 13	3, 2, 3	7, 7, 5	1, 2, 2

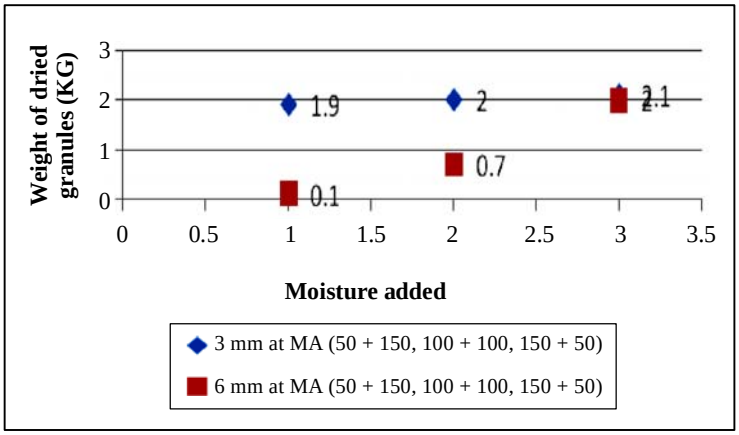

Fig. 12: Weight of different size of dried granules (3 mm & 6-10) mm vs moiture added (MA)

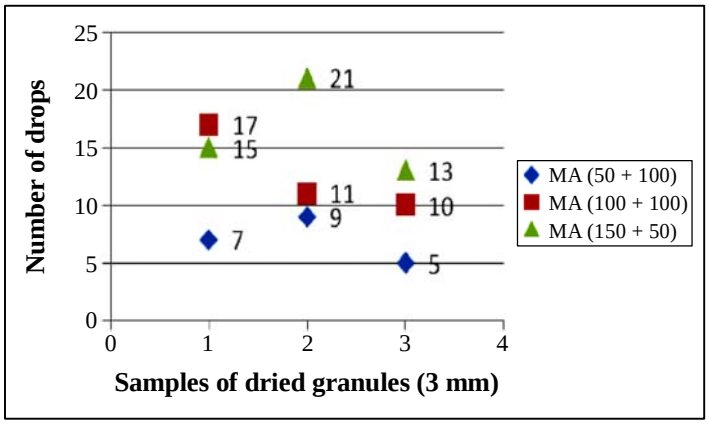

Fig. 13: Number of drops from 45 cm vs samples of dried granules (3 mm)

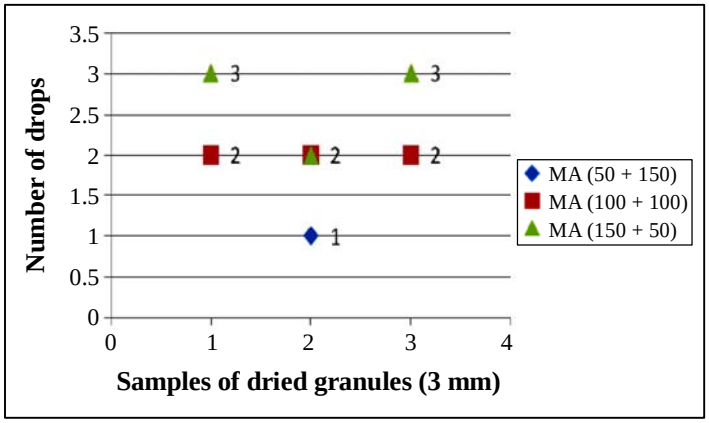

Fig. 14: Number of drops from 180 cm vs samples of dried granules (3 mm)

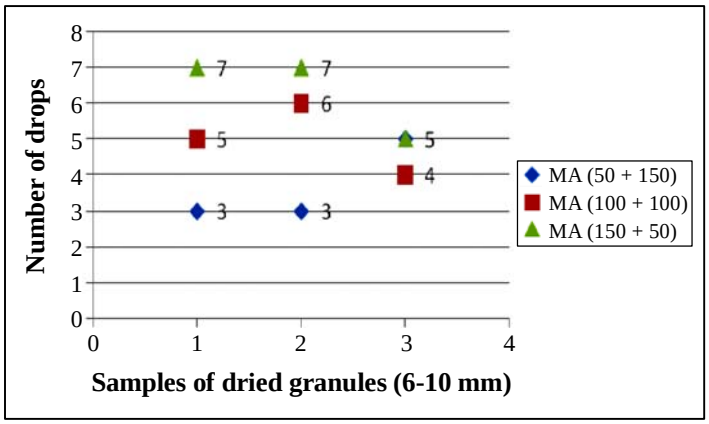

Fig. 15: Number of drops from 45 cm vs samples of dried granules (6-10 mm)

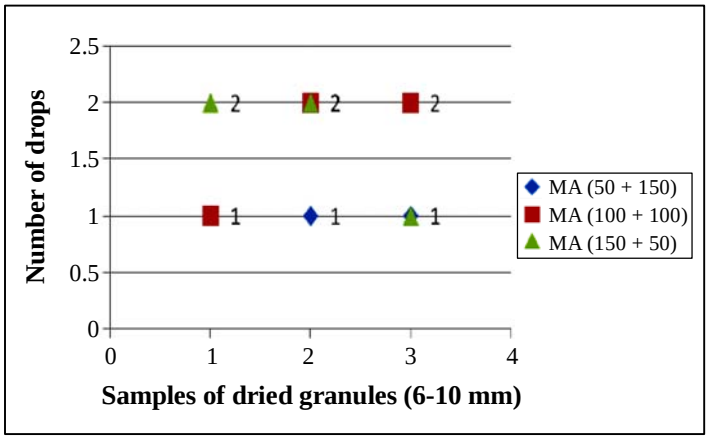

Fig. 16: Number of drops from 180 cm vs samples of dried granules (6-10 mm)

Number of droppings is decreased after coking as shown in Figs. 13-16. It determines strength of granules is decreased after coking when compared to before coking due to removal of liquid binding in coking.

CONCLUSION

As increasing in moisture content leads to increase in granule formation. The moisture added in first time interval helps in nucleation and moisture added in second time interval helps in layering and coalescence. In MA (50 + 150) condition there is lack of nucleation of granules due to insufficient moisture content in first interval. In MA (100 + 100) condition there is an over layering and abrasion transfer leads to large granules in second interval. In MA (150 + 50) condition is an optimum condition of proper nucleation, layering and coalescence, which is done in two intervals.

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