

**An Investigation on Seeded Granules Formation in a
Continuous Granulator using Discrete Element Method**

by

Nur Fardina binti Abd Ghani

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

MAY 2012

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

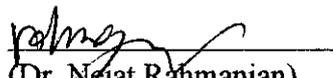
An Investigation on Seeded Granules Formation in a Continuous Granulator using Discrete Element Method

by

Nur Fardina binti Abd Ghani

A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHCEMICAL ENGINEERING)

Approved by,


(Dr. Nejat Rahmanian)
Supervisor.

Universiti Teknologi PETRONAS

TRONOH, PERAK

May 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



NUR FARDINA BINTI ABD GHANI

ABSTRACT

Consistency in production of granules is crucial and hard, as not much is known about solid processing as compared to fluid mixing. Considerable efforts have been made in the past years to gain fundamental understanding in this matter but most researchers have seen to be interested in the batch process, as it offers advantages with respect to quality assurance. This is none other than due to a batch can always be accepted or rejected. However, obviously continuous process offers lots of advantages too and one of them is that they are a more reliable option for the large scale industry. Thus purpose of this study is to get a better understanding of the granulation process in a continuous mixer type of granulator. Discrete Element Method (EDEM) simulation is to be performed to look into the particle dynamics on the scale of individual particles.

ACKNOWLEDGEMENT

First and foremost, Alhamdulillah and thank you Allah the Almighty for guiding me throughout this journey in completing this Final Year Project (FYP). Submission of this Dissertation will mark my last effort in completing this five-year Bachelor's degree studies and I hope this project will be useful for the future betterment of solid processing industry.

A token of appreciation goes to my supervisor Dr. Nejat Rahmanian for his valuable guidance and advice throughout the progress of this project. His passion in Particle Technology and Solid Processing Course has been really inspiring and helpful for me. I got to learn a lot not only from him, but he also referred me to his colleague in University of Leeds UK, Dr. Ali Hassanpour and it was an honour to learn from these great people.

Besides that, I would also like to thank all lecturers in Chemical Engineering Department of Universiti Teknologi PETRONAS (UTP) for any direct or non-direct help provided in completing this project. Finally, I would like to express my gratitude to all my colleagues from the same intake for their continuous support and encouragements.

TABLE OF CONTENTS

| | |
|--|------|
| Verification Statement | i |
| Abstract | iii |
| Acknowledgments | iv |
| Table of Contents..... | v |
| List of Figures..... | vii |
| List of Tables..... | viii |
| 1. INTRODUCTION | |
| 1.1 Background Study | 1 |
| 1.2 Problem Statement..... | 2 |
| 1.3 Objectives | 2 |
| 1.4 Scope of Study..... | 3 |
| 2. LITERATURE REVIEW AND THEORY | |
| 2.1 Background of Granulation Process..... | 4 |
| 2.2 Seeded Granulation..... | 5 |
| 2.3 Discrete Element Method (DEM) | 6 |
| 2.4 Governing Equations and Force Models | 7 |
| 2.4.1 Approaches in DEM..... | 7 |
| 2.4.2 Motion of Particles | 8 |
| 2.4.3 Contact Forces Between Particles..... | 9 |
| 2.5 Flow Pattern in Rotating Drums..... | 12 |

| | | |
|-------|---|----|
| 3. | METHODOLOGY / PROJECT WORK | |
| 3.1 | Research Methodology | 13 |
| 3.1.1 | Literature Review Survey | 14 |
| 3.1.2 | Software Familiarization | 14 |
| 3.2 | Project Work..... | 15 |
| 3.2.1 | Contact Model Applied | 15 |
| 3.2.2 | Simulation Set Parameters..... | 16 |
| 3.2.3 | Drum Granulator Model | 17 |
| 3.3 | Key Milestones | 17 |
| 3.4 | Gantt Chart | 18 |
| 4. | RESULTS AND DISCUSSION | |
| 4.1 | Effect of Granulator Design | 21 |
| 4.2 | Effect of Drum Rotational Speed | 22 |
| 4.3 | Effect of Particle's Surface Energy | 22 |
| 4.4 | Effect of Seed/ Fine Particles Size Ratio..... | 23 |
| 5. | CONCLUSION..... | 24 |
| 5.1 | Future Work Recommendation | 25 |
| | REFERENCES | 26 |
| | APPENDIX I | |
| | (Paper Published and Accepted for Oral Presentation in 5th Asian Particle Technology 2012, held in National University of Singapore 2-5th July 2012..... | 28 |

LIST OF FIGURES

Chapter 2

1. SEM image of seeded granules produced experimentally 5
2. Regime map presented for production of seeded granule structure in Cyclomix granulator 6
3. Coordinates system used to describe collision model in DEM 10
4. Illustration of the difference between two most used contact models 11
5. EDEM JKR as a combination of Hertz-Mindlin and JKR Theory 11
6. Time-averaged spatial distribution in a rotating drum 12

Chapter 3

7. Methodology used by EDEM during simulation 15
8. Front and side view of model granulator 17

Chapter 4

9. Snapshot of simulation progress at sequential simulation time 19
10. Snapshot of simulation progress at 5 sec for different rotational speed 20
11. Snapshot of several cases where fines surrounding seed particle 20
12. Cross sectional view of inside the drum granulator 21
13. Isometric view of drum granulator designed with scoops 21
14. Potential seeded granules at different granulator design 22
15. Potential seeded granules at different drum rotational speed 23
16. Potential seeded granules at different particle's surface energy 23

LIST OF TABLES

Chapter 3

| | |
|---|----|
| 1. Set parameters input to simulation | 16 |
| 2. Interactional properties used in the simulation | 16 |
| 3. Dimensions used for model granulator without hoppers | 17 |
| 4. Expected key milestone | 17 |
| 5. Project timeline for FYP 1 | 18 |
| 6. Project timeline for FYP 2 | 18 |

CHAPTER 1

INTRODUCTION

1.1 Background Study

Granulation process, simply defined as particle enlargement is a process of collecting particles together by creating bonds between them, and the bonds can be formed by compression or using binding agent (Tousey, 2002). There is several obvious importance of these granules formation, and amongst them is to improve material properties and performance such as increasing flow ability, easy handling and to reduce dustiness.

Consistency in production of granules is crucial and hard, as not much is known about solid processing as compared to fluid mixing. For example, the slightest change in content's properties or process parameters for manufacturing of drugs can have a huge impact on its quality and a person's health. Is it thus important to understand the physical phenomena that govern the flow and mixing of solid ingredients involved in the manufacturing of granules product, as for example; the pharmaceuticals industry.

Thus nowadays, there are increasing numbers of researches done focused on the study of factors leading to consistency in granules production industry. Despite the success in experiments and simulations conducted for the batch, now it is time to look more into continuous process as it can overcome certain points that batch process are lacking, and in the same time they offer numerous advantages too.

1.2 Problem Statement

Batch process in granules production industries undeniably offers many advantages with respect to quality assurance because a batch can always be accepted or rejected. However so, due to limitations of batch process in scale up and the urge in large scale production, continuous process has been seen as a suitable replacement. Hence the aim of this research is to better understand the flow and mixing behavior within the designed continuous granulator using simulation technique. Seeded granules formation has been the main focused in this study. Formulation of parameters was performed to study the characteristics in producing the same quality product as in the batch method.

1.3 Objectives

This research project focuses on the following objectives:

1. To perform simulations of continuous rotating drum granulator by applying Discrete Element Method (DEM).
2. To study the effect of formulation parameters such as drum rotational speed, and particle's parameters and properties on seeded granules formation.
3. To study the effect of operating conditions on flow and mixing behaviour

1.4 Scope of Study

The scope of the study is to get a better understanding of granulation process using the Discrete Element Method, in which it is modelled in a continuous granulator. With the aim to improve continuous granulation process through the ability of producing consistent products, standardized parameters and operating condition of the granulation process is to be analyzed. Drum granulator has been chosen to be the model geometry for this study for the simple and ease of design.

In order to do the simulation works, fundamentals of granulation process and particulate characteristics need to be studied first. Basic discrete element framework and how particle interact to each other (contact mechanisms) are among the key point in understanding granule behaviours in granulator. Granule's behaviours such as flow patterns and the suitable operating conditions will be determined from the simulation results.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Background of Granulation Process

Granulation technology is widely used in numerous manufacturing industries such as production of food, fertilizers, pharmaceutical in tablets and so on. There are numbers of techniques available to combine these seeds and fine particles into required forms, and the most well known of them are wet, dry and melt granulation technique. As for this research project, the study will be focusing on the wet granulation method in a continuous rotating drum granulator.

Batch process in granules production industries undeniably offers many advantages with respect to quality assurance because a batch can always be accepted or rejected. However, one of the most vital challenges for batch process is process scale-up. Considerable efforts have been made in the past years to gain fundamental understanding of scale up characteristics and the effects on granules (Rahmanian *et al.*, 2008; Hassanpour *et al.*, 2008). More researchers have also tried to avoid the scale up problems by evaluating other alternatives of the batch production. Continuous or semi-continuous processes are amongst the promising choices.

Thus nowadays, there is increasing numbers of studies regarding the replacement of batch with the continuous method (Betz *et al.*, 2003; Vervaeet and Remon, 2005). As for this Final Year Project, the author is focusing on the simulation of continuous granulator to see the flow and mixing behaviours of granules due to different operating parameters. Software Extended Discrete Element Method (EDEM Ver. 2.4) is the most suitable approach to describe the details on the scale of individual particulate.

2.2 Seeded Granulation

Well said in a review journal by (Zhu *et al.*, 2007); macroscopic behaviour of particulate matter is mostly controlled by its microscopic mechanism. Thus, more has realized that not only external factor such as equipment control strategies that govern the behaviour and properties of granules, but most importantly is the internal structure and mechanisms leading to the difference that matter.

In their journal, (Rahmanian *et al.*, 2011) described one method of granulation believed to assure consistencies in granulation termed as ‘seeded granulation’ technique. This seeding mechanism occurs when larger feed particle usually known as seed being a nucleus at the granule core and partially wetted fine particles and seeds adhere to each other forming granules as can be seen in Figure 1.



Figure 1 SEM image of seeded granules produced in experiment conducted by (Rahmanian and Ghadiri, 2011)

It was found that the factors strongly affecting and giving rise to formation of seeded granules in high shear mixer is optimum impeller tip speed and primary particle size distribution. A regime map of conditions preferable for seeded granules formation has been established for the batch granulator model of Cyclomix as in Figure 2.

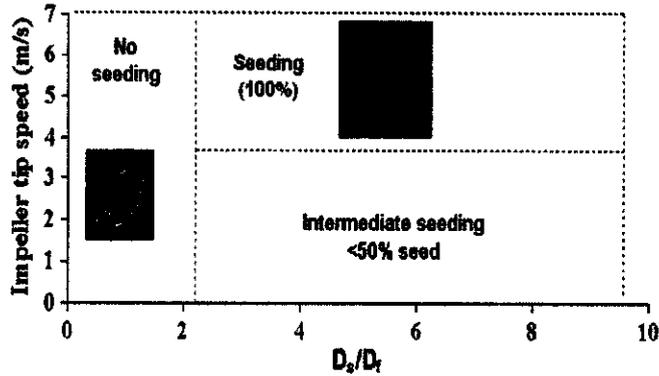


Figure 2 Regime map presented for production of seeded granule structure in Cyclomix granulator (Rahmanian *et al.*, 2011)

Continuing from that, it is highly desirable to further analyse the cause of seeded granulation and process parameters leading to it and this time, in a continuous granulator.

2.3 Discrete Element Method (DEM)

In recent years, simulation method for discrete particle has been rapidly developed worldwide, mainly as the result of rapid development of discrete particle modelling technique and also computer/ software expertise. Several techniques of this particulate simulation have been developed, including Monte Carlo method, cellular automata and discrete element method (DEM). Simulation work to be used in this research is based on the DEM approach widely used in the literature to predict flows of solid-particulate materials.

Believing the concept that macroscopic behaviour of particulate matter is controlled by its microscopic mechanism, this DEM models granular medium as a collection of particles whose momentum and forces are studied separately. When particles collide with each other, a local constitutive law determines the inter-particle contact force and resulting motions of the particles involved. Understanding of this microscopic aspect of particle's movement or reaction is the key point of reliable research and into producing dependable results. This aim can only be achieved via particle scale research based on detailed micro dynamic information.

There is large bulk volume of publications relating to DEM have been published in the past decades or so, however these two review papers had been really helpful for the author as it covers almost all the history and recent inventions on the topics (Zhu *et al.*, 2007; Zhu *et al.*, 2008). As early proposed by Cundall in 1979, the governing equations are a set of equilibrium equations for resultant forces and moments at centres of particles (Cundall, 1979). These forces emerge from contact forces, body forces, inertia forces and damping forces. Since then, the DEM method has been widely applied to various fields such as soil mechanics, material science and processing industry.

2.4 Governing Equations and Force Models

2.4.1 Approaches in DEM

Two types of DEMs are most common; soft-particle and hard-particle approaches (Zhu *et al.*, 2007). As stated earlier, Cundall's method was the first granular dynamics simulation technique published in the open literature. This method is one kind of soft-sphere method where particles go through slight deformations, and these moments are used to calculate elastic, plastic and frictional forces between particles. Newton's law of motion is used to estimate the next motion of the particles. Advantages of this soft-style approaches is that, they are capable of handling multiple particle contact simultaneously which is important when modelling quasi-static system.

Quite the opposite of the above method; for the hard-style approaches, collisions of particles are assumed to occur one at a time, instantaneous and must not occur simultaneously. Typically this method is most useful in rapid granular flows. As for this particular study, soft-sphere method is basically applied to see the multiple collisions occurring on a single particle.

2.4.2 Motion of Particles

A particle in a granular flow can have two types of motion; translational and rotational. Along its movement, the particle may interact with its neighbouring particle, walls or surrounding fluid through which the momentum and energy are exchanged. For the real case scenario, this movement is not affected only by the forces and torques of neighbouring particles and vicinal fluid, but also from the particles and fluids far away through propagation of disturbance waves.

To overcome this complexity of such calculation process, in DEM, it is generally assumed that this problem can be resolved by choosing a numerical time step less than a critical value. Even though this will consume more computational time, but during this shorter time step, disturbance cannot propagate from the particle and fluid farther than its immediate neighbouring particles and vicinal fluid. For example as in this simulation work, fixed time step was set at a very small value, which is 1.26×10^{-5} second. Thus at all times, resultant force and motion of a granule/ particle can be determined wholly from its interaction with only the nearby fine particles and fluids.

However specifically for this research paper, the author focused only on particle modelling, and does not coupled with fluid movement (CFD software). It is assumed that particle is already cohesive and binders are distributed evenly amongst particles. Hence based on these considerations, Newton's second law of motion can be used to describe the motion of individual particles, with excluding the vicinal fluid. The governing equations for estimating motion of particles can be written as:

$$m_i \frac{d\mathbf{v}_i}{dt} = \sum_j F_{ij}^c + \sum_k F_{ij}^{nc} + F_i^f + F_i^g$$

$$I_i \frac{d\omega_i}{dt} = \sum_j M_{ij}$$

Various models have been proposed and developed to calculate these contact forces F_{ij}^c and non-contact forces F_{ij}^{nc} , which will be discussed in further section.

2.4.3 Contact Forces between Particles

In case of collision between two particles, contact force in DEM models are commonly calculated according to a soft-sphere contact model based on theory developed by (Hertz, 1882) for the normal impact. This theory relates the circular contact area of a sphere with a plane (or could be a sphere too) to elastic deformation properties of materials and it neglects any surface interactions in its presumption.

The force-displacement relation of two particles in normal direction is solved by using the following equations;

$$k_n = M\sqrt{n}$$

in which:

$$M = \frac{2\sqrt{2r}G_\mu}{3(1-\nu_\mu)} \quad \text{and} \quad n = r_i + r_j - d \quad \text{and} \quad r = \frac{2r_i r_j}{r_i + r_j}$$

The particle stiffness M depends on the shear modulus G_μ and the Poisson's ratio ν_μ of the material of particle, also on the average size of both particles ($r_i + r_j$).

A no-slip approximation of the model by (Mindlin and Deresiewicz, 1953), on the other hand is used for the tangential component of the contact force, as proposed by (Tsuji *et al.*, 1992). The shear force F_s is proportional to the shear displacement s_h for the elastic area.

$$F_s = k_s s_h$$

The stiffness in shear direction k_s can be related to the stiffness in normal direction:

$$k_s = k_v k_n$$

in which:

$$k_v = 3 \frac{1-\nu_\mu}{2-\nu_\mu}$$

This means that the relation between stiffness's of the normal and tangential direction depends only on the Poisson's ratio of material used for the particles/ granules.

Figure 3 summarizes the coordinate system commonly used in the description of the collision model (Deen *et al.*, 2007). \mathbf{n}_{ab} in the figure represents normal contact force of the collisions, while \mathbf{t}_{ab} represents tangential component of the contact forces.

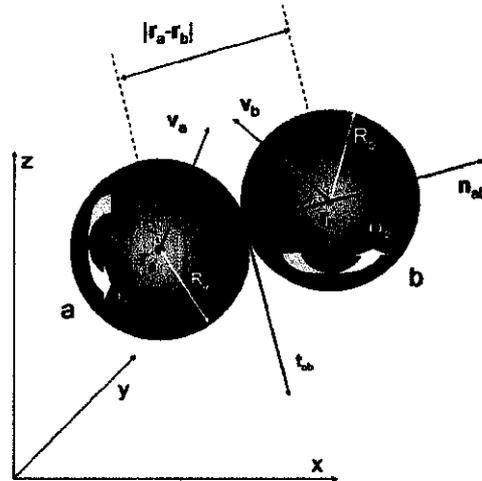


Figure 3 Coordinate system used to describe collision model in DEM (Deen *et al.*, 2007).

As mentioned above, the classical Hertz contact theory is precisely for elastic deformation of bodies in contact, and it neglects adhesion force due to van der Waals attraction. To improve this theory, there are two commonly used models developed named as the JKR (Johnson *et al.*, 1971) and DMT (Derjaguin *et al.*, 1975) models which were invented in a way that both theories consider adhesion force in their assumption.

DMT model handles Hertz deformation and adhesion effect separately, whilst in JKR theory, contact between particles is considered to be adhesive hence it correlates the contact area to the elastic material properties plus the interfacial interaction strength. JKR were modelled to recognize that both tensile and compressive interactions contribute to the total contact radius, and basically this theory is to be used as the basic contact mechanism in this simulations work.

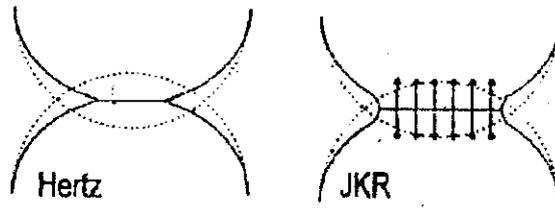


Figure 4 Illustration of the difference between two most used particle contact models (Contact Mechanics, 2012)

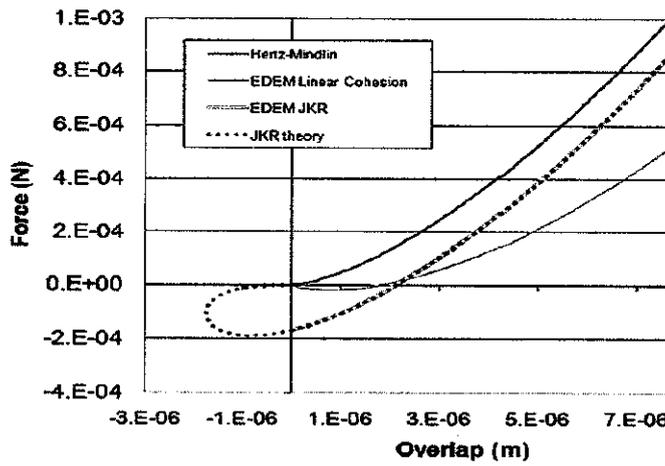


Figure 5 EDEM JKR as a combination of Hertz-Mindlin concept and JKR Theory (Online training by DEM Solutions, UK)

2.5 Flow Pattern in Rotating Drums

Drums are well known as one of common devices in industry for mixing, drying, milling, coating and granulation. Particle flow in rotating drums varies and exhibits a range of complex phenomena such as avalanche, segregation and convection. This has triggered considerable interest among researchers, hence over the past decades; many research works mainly at macroscopic level have been done in this area.

Basically, six flow regimes have been observed in the transverse direction over a range of rotational speeds (Henein et al., 1983). Some of the regimes have been reproduced by DEM modelling and several agreements are made between experimental and modelling works. For example, it has been observed that at low speed rotation, avalanches tend to occur at the surface of different size particles with a constant angular velocity. Meanwhile as for in the rolling regime, flows of particles in rotating drum will usually produce even surface of granular materials.

Micro dynamic structure of particles in drum was the analysed by (Yang *et al.*, 2003). Based on the DEM results, both spatial and statistical distributions of micro dynamic variables related to flow and force structure has been established (Figure 6). The research team demonstrated that resulting micro dynamic information is useful in understanding the observed effect of rotation speed on agglomeration. This is in the agreement with this FYP study to investigate seeded granules formation, in a way that the author manipulated rotation speed to see the effect on the agglomerates produced.

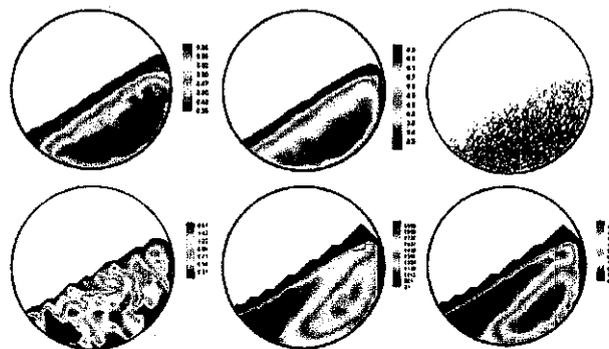


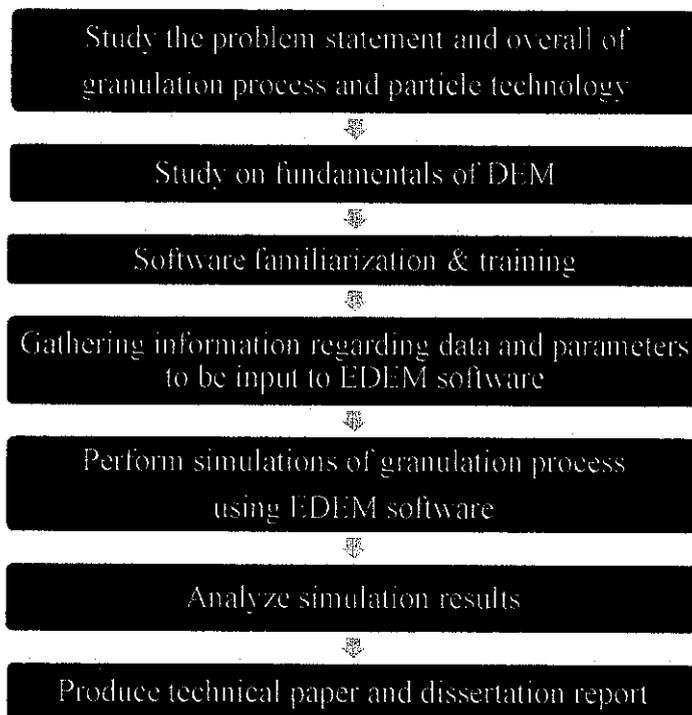
Figure 6 Time-averaged spatial distribution of (from left to right and top to bottom): porosity, coordination number, (snapshot) force network, total force (mg), collisions velocity (m/s), and collision frequency (1/s) in a rotating drum at speed 20 rpm (Yang *et al.*, 2003)

CHAPTER 3

METHODOLOGY/ PROJECT WORK

3.1 Research Methodology

This methodology covers the whole two semester's research work.



3.1.1 Literature Review Survey

This literature review survey and study has been done starting from the beginning of research period, and continued towards the end of first semester study. The author has covered the overall aspect of the research studies such as;

- i) Particle Technology,
- ii) Contact Mechanics,
- iii) Seeded Granulation, and
- iv) Discrete Element Method.

3.1.2 Software Familiarization

Software used in this research study is EDEM Ver. 2.4 and this software for the first time was launched in Malaysia aiming to establish a centre of Powder Technology in UTP. For the software familiarization, the author had participated in two official online training sessions conducted by DEM Solutions Ltd., United Kingdom.

Both sessions cover topics of basic manuals on how to use the software, the mathematics behind it, contact mechanics available to be applied and a brief discussions regarding modelling and simulations technique. The training was with guidance of supervisor, Dr. Nejat Rahmanian and also Dr. Nurul Hassan. Follow up training sessions were also performed with other UTP Master and PhD students using the tutorials and examples as prepared by the same company.

3.2 Project Work

3.2.1 Contact Model Applied

There are numbers of contact models available in EDEM software and basically all these models describe how elements behave when they come into contact. The behaviour of individual elements affects bulk behaviour of the system; hence it is crucial in determining which model is best fit for our case studies.

As for this simulation work, particle's contact model applied was the Hertz-Mindlin with JKR model, an improved version in EDEM, but with similar fundamental to what has been used by (Hassanpour *et al.*,2011). Referring to what has been stated in the journal of seeding in the batch granulator, Hassanpour and his team applied two contact models simultaneously during simulation which are Hertz-Mindlin model and also linear cohesion model.

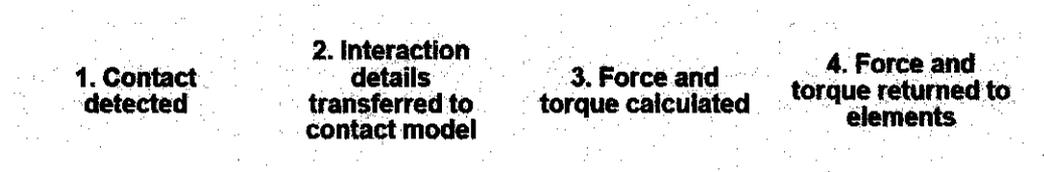


Figure 7 Methodology used by EDEM during simulation.
(DEM Solution UK, 2012)

Hertz-Mindlin theory is basically developed in case of collision between two particles; where Hertz theory calculates contact forces for the normal impact and Mindlin for the tangential component of the normal force. Any surface interactions such as Van Der Waals or contact adhesive interactions are neglected in this theory.

Hence to simulate the cohesion force between particles, in which in this study we assume to use Polyethylene Glycol (PEG) 4000 as the binder; another complimentary contact model is needed. This cohesive occurrence can be set by applying either linear cohesion model or JKR model. JKR Theory in EDEM is the recent contact model by DEM Solutions UK and a better fit for our studies. Value of cohesion energy density used is calculated based on a surface energy from the JKR theory, and as for this work the value used as input of interface energy between particles is 1.5 J/m^2 (Hassanpour *et al.*,2011).

3.2.2 Simulation Set Parameters

The seeds and fine particles' parameters input to this simulation is set to be similar according to experimental characteristics of Calcium Carbonate powder and Polyethylene Glycol (PEG) 4000 as the binder. These input values were referred from journal of seeded granulation study by (Rahmanian *et al.*, 2011; Hassanpour *et al.*, 2011). Both studies and the previous publications by this group of researchers have focused on the granulation mechanism in a batch granulator.

Table 1 Set parameters input to simulation

| Parameter | Particles | | Geometry |
|-------------------------------|-----------|------|----------|
| Density (kg.m ⁻³) | 800 | | 1200 |
| Poisson's ratio | 0.2 | | 0.5 |
| Young's modulus (GPa) | 0.24 | | 0.003 |
| | Seeds | | Fines |
| Diameter (mm) | 8 | 2 | - |
| Production (s ⁻¹) | 40 | 4000 | - |

As for the interactional properties used, the values are based on cohesive resembling of particles where it is assumed that binder is uniformly distributed among all particles.

Table 2 Interactional properties used in the simulation

| Interaction | Particle- Particle | Particle – Geometry wall |
|---------------------------------|-----------------------|-----------------------------|
| Coefficient of restitution | 0.1 | 0.1 |
| Coefficient of static friction | 0.5 | 0.2 |
| Coefficient of rolling friction | 0.002 | 0.002 |

3.2.3 Drum Granulator Model

As for the granulator model, the geometry of drum granulator was designed using AutoCAD 2007 software and exported to the EDEM Ver. 2.4 software (DEM Solutions, UK). Table 3 list parameters and dimensions which were used to design model, while the model granulators are shown in Figure 8.

Table 3 Dimensions used for model granulator without hoppers

| Parameter | Value |
|----------------------------|---------|
| Volume of granulator (L) | ~ 30 |
| Length (mm) | 800 |
| Outer/ Inner Diameter (mm) | 200/199 |
| Gradient angle (°) | 2 |

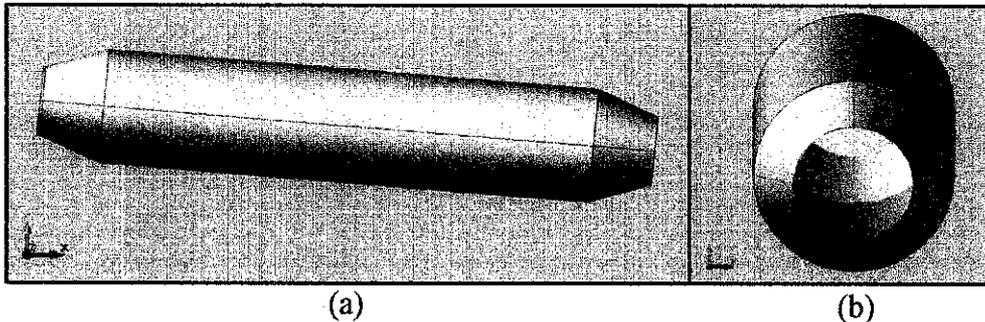


Figure 8 (a) Right side view of the granulator. (b) Front view

3.3 Key Milestones

Table 4 shows the scheduled due date for submission of major events in Final Year Project 2 course.

Table 4 Expected key milestone

| Milestone | Expected Completion Date |
|--|--------------------------|
| Drum granulator process simulations | 30/7/2012 |
| Submission of technical paper | 15/8/2012 |
| Oral presentation | 27/8/2012 |
| Submission of hard bound dissertation report | 16/9/2012 |

3.4 Gantt Chart

Table 5 Project Timeline for FYP 1

| Activities | FYP 1 -Week Number | | | | | | | | | | | | | |
|--------------------------------------|--------------------|---|---|---|---|---|---|---|---|----|----|----|----|----|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Topic Selection | █ | █ | | | | | | | | | | | | |
| Research About Topic | | █ | █ | █ | █ | | | | | | | | | |
| Complete Literature Review | | | █ | █ | █ | █ | █ | █ | █ | █ | █ | | | |
| Submission of Extended Proposal | | | | | | █ | █ | | | | | | | |
| Proposal Defense | | | | | | | | █ | █ | | | | | |
| Software Familiarization | | | | | | █ | █ | █ | █ | | | | | |
| Software Online Training | | | | | | | | █ | █ | | | | | |
| Material and Geometry Identification | | | | | | | | | █ | █ | █ | | | |
| Simulation Work and Data Analysis 1 | | | | | | | | | | █ | █ | █ | | |
| Submission of Interim Draft Report | | | | | | | | | | | | █ | █ | |
| Submission of Interim Report | | | | | | | | | | | | | | █ |

Table 6 Project timeline for FYP 2

| Activities | FYP 2-Week Number | | | | | | | | | | | | | |
|---|-------------------|---|---|---|---|---|---|---|---|----|----|----|----|----|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Simulation Work and Data Analysis | █ | █ | █ | █ | █ | █ | █ | █ | █ | █ | | | | |
| Preparation of slide presentation for APT 2012, Singapore | | | | | █ | █ | | | | | | | | |
| Submission of Progress Report | | | | | | | █ | █ | | | | | | |
| Final Data Analysis and Report | | | | | | | | █ | █ | █ | | | | |
| Submission of Draft Dissertation Report | | | | | | | | | | | █ | █ | | |
| Submission of Dissertation and Technical Paper | | | | | | | | | | | | █ | █ | |
| Oral Presentation | | | | | | | | | | | | | █ | █ |
| Submission of Project Dissertation | | | | | | | | | | | | | █ | █ |

CHAPTER 4

RESULTS AND DISCUSSION

This simulation was carried out by introducing constant size of seed and fine particles at the left end of drum granulator with aiming to produce seeded granules. Particles were let to settle under natural gravity force and this filling works simultaneously with rotation of the drum. This is set as to demonstrate continuous processing environment where process occur simultaneously when particles being fed into and coming out from the system. Simulation was then carried out for 10 s real time at three different rotational speeds; 10, 50 and 100 rpm.

Figure 9 shows the snapshot of simulation progress at sequential simulation time (0, 3, 6, 9 s), with drum rotational speed at 10 rpm and particle surface energy of 1.5 J/m^2 . The figures show us that especially for this set of parameters, most of the fines settled at the bottom of drum, whilst seed particles in red colour were either on top of those 2 mm particles or in the middle of it. Segregation has been observed in this simulation, as opposed to the desired agglomeration. However, this is believed to occur due to the lack of adhesion force between particles.

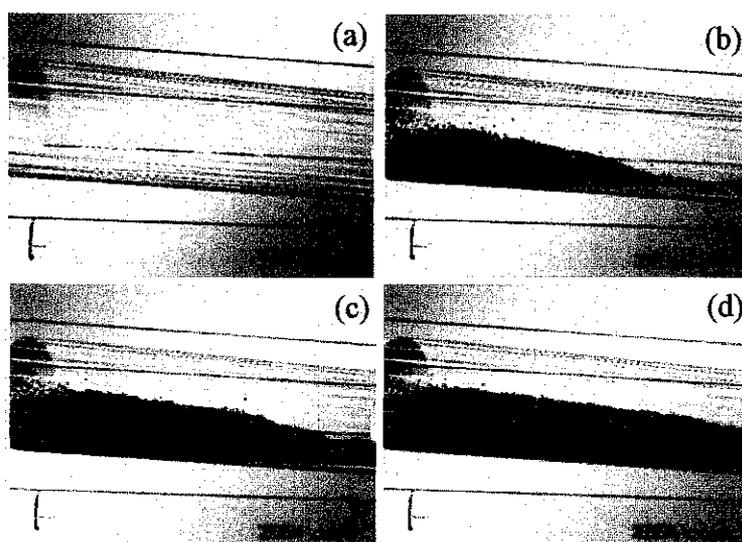


Figure 9 Snapshot of simulation progress at sequential simulation time
(a) 0s (b) 3s (c) 6s (d) 9s

Throughout simulation progress for all cases, it was clear that seeds and fines have collided and in contact with each other, but no fully covered agglomerate was sure to have formed. The clearest result can be seen in Figure 10 where the figures show the zoomed in snapshot of particles during simulation progress. Interaction in the form of contact vector between seed and fine particles is shown in these figures as a straight line connecting the particles, as highlighted in purple colour. Hence it is clear that bonds and contacts between seeds and the surrounding fine particles exist, and we termed this as potential seeded granules.

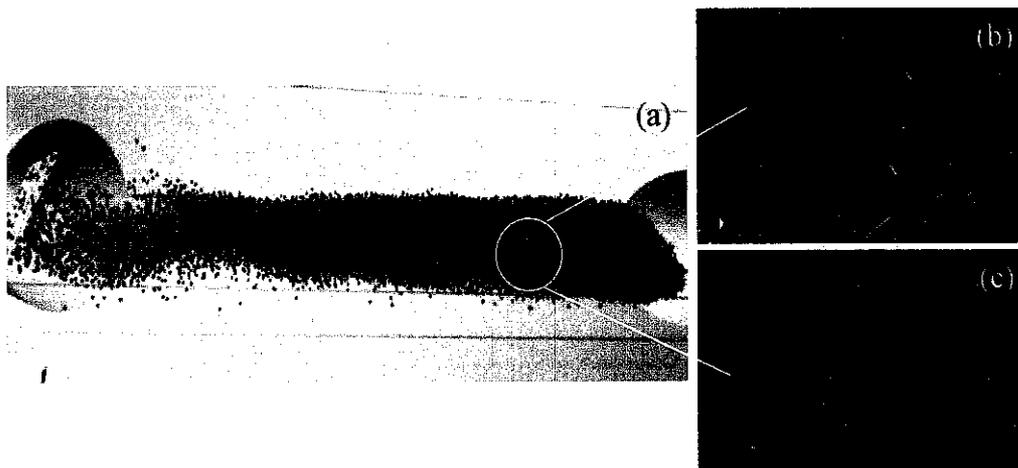


Figure 10 Snapshot of simulation progress at 5s for the 10 rpm rotational speed.

- (a) Overall view from the top side of the drum
- (b) Snapshot focusing on several numbers of seeds.
- (c) A clearer snapshot of one seed and its connection with neighbouring fines.

Figure 11 (a) and (b) show the pictures of only one seed each and its fines coverage (other particles were not displayed for clarity). In theory, a properly seeded granule should have the seed fully covered with at least one layer of fine particles. Based on the size ratio for this case, $d_{seed}/d_{fine}=4$, a seed should be in contact with approximately 50 fine particles to get full coverage.

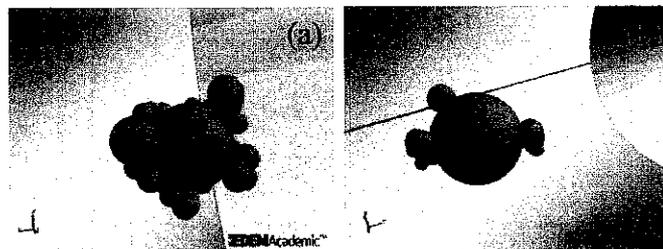


Figure 11 Snapshot of two cases where fines covering one seed particle, with about (a) 40% coverage (b) 10% coverage.

However, a more detailed and precise computer algorithm should be developed to know all seeds percentage coverage of fines instead of just a manual estimation and observation. In the next sub section, further analysis of these simulation work was done accordingly by manipulating several variables such as drum rotational speed and particle's surface energy.

4.1 Effect of granulator design

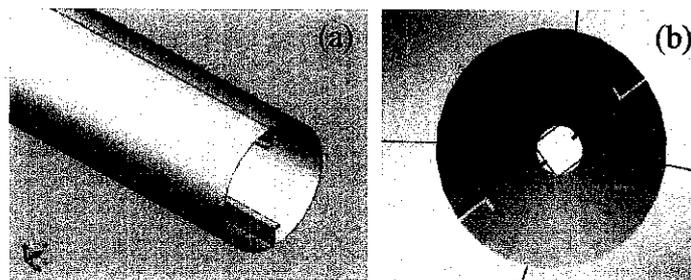


Figure 12 Drum granulator designed with scoops. (a) Isometric view (b) Front view

The simulation work was then performed using a different design of granulator, in which scoops are placed at the inner side of the drum (Figure 12). As shown in the figure, drum with scoops produce a more turbulent motion of particles and this indirectly create a rapid collisions and contacts between seeds and fines. This type of motion has actually leads to the rapid growth and breakage of the agglomerates. Nevertheless, at the end of the simulation, there is not much difference between total potential numbers of seeded granules over time, between both designs.

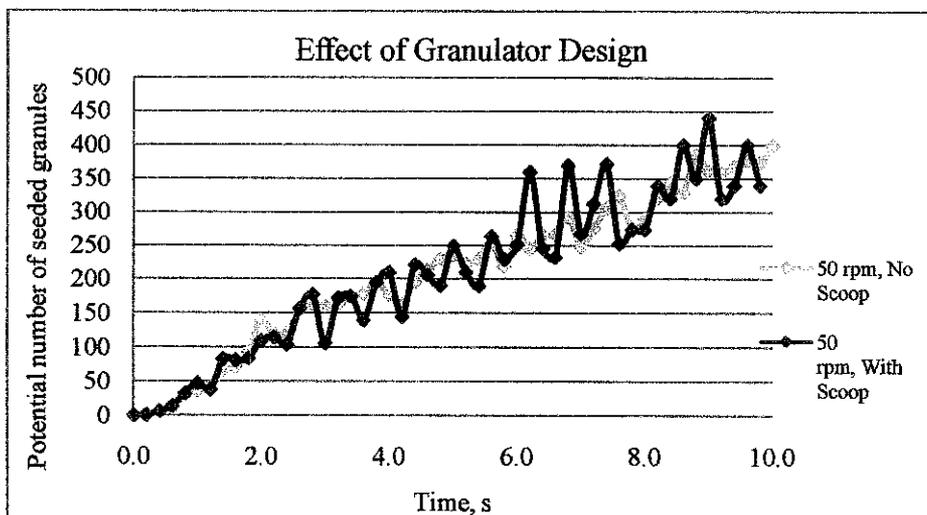


Figure 13 Potential seeded granules at different granulator design

4.2 Effect of drum rotational speed

Figure 14 shows that potential number of seeded granules is higher in a low speed rotating drum as compared to a high speed rotating drum. This result is in agreement with the findings of (Hassanpour *et al.*, 2012) in their work in the batch granulator, that seeded granules are formed with a higher probability at low rotational speed of model.

However, it should be noted that for the results in these variable studies, the potential seeded granules are taken as those which have a seed contacting with fines, regardless of its surface coverage. This means that even with sparse coverage of a seed, it is still counted as a potential seeded granule in this study.

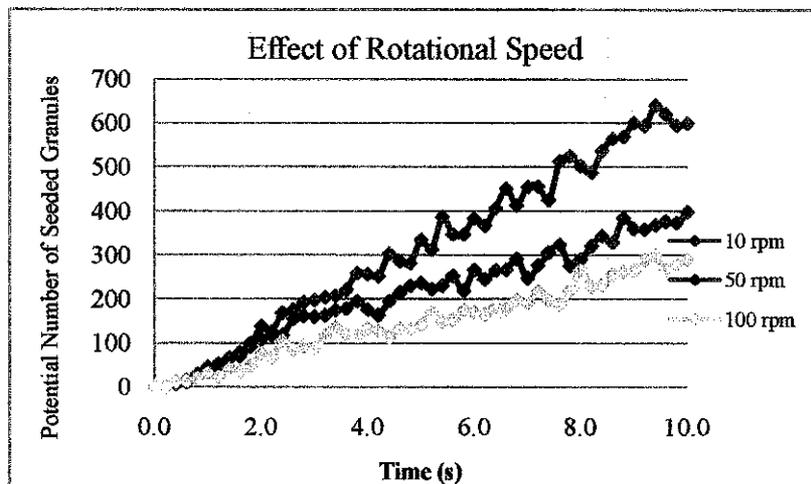


Figure 14 Potential seeded granules at different drum rotational speed, using model granulator without scoops.

4.3 Effect of particle's surface energy

JKR Theory stated that after compression, particles are not going back to their original position because of a surface tension force. In the JKR model in EDEM, this force is dependent on the area formed by overlap and a surface energy value. We believed that a greater value of particle's surface energy will give a higher cohesion impact between particles, thus a higher numbers of potential seeded granules formation.

However as a result of manipulating value of surface energy (Figure 15), it was proven that higher value of surface energy of particles will produce a condition of

less contact and collision between seed and fine particles. This might have occurred due to the higher surface energy and surface tension force of particles, leading to high impact collision, but less chaotic movement of overall system. In other words, higher surface energy of particles produced less seeded granules, but probably higher percentage coverage of each seed.

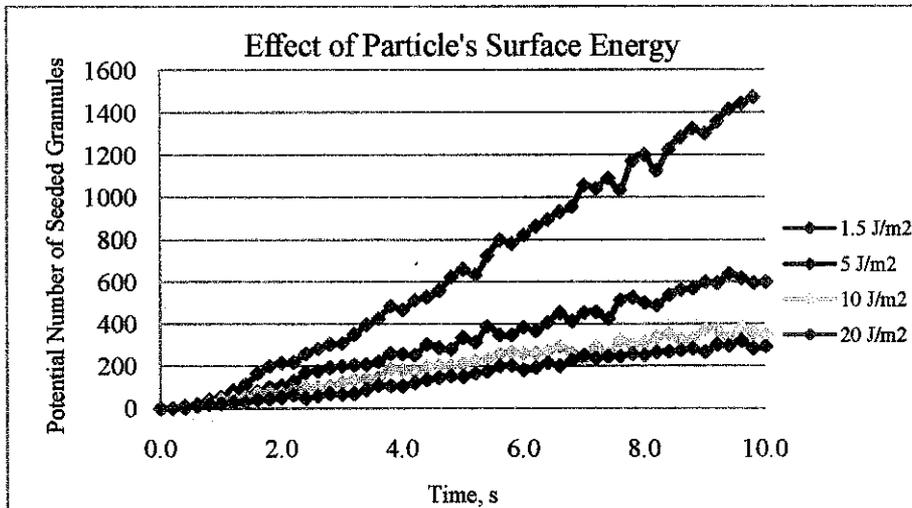


Figure 15 Potential seeded granules at different surface energy.

4.4 Effect of seed/ fines size ratio

Figure 16 shows the effect of different size ratio of input particles to simulation. It is concluded that a higher ratio of the sizes will have a higher probability of producing seeded granules.

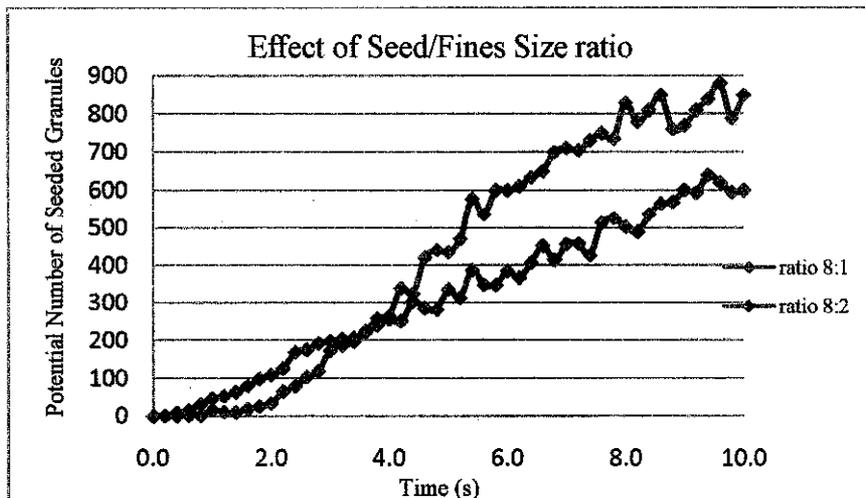


Figure 16 Potential seeded granules at different seed/fines size ratio.

Further refinement of these results is required to provide a more realistic criterion for defining conditions for seeded granules formation.

CHAPTER 5

CONCLUSION

This study is part of an ongoing research to get a better understanding of the seeded granules formation in a continuous granulation process. This project is important in such a way that continuous process could replace batch process in many applications with aiming in large production of granules with consistent physical and mechanical properties. Drum granulator was used as the model of the continuous process for this Final Year Project. Simulation using DEM was carried out to look into the particle dynamics on the scale of each individual particles and granules.

On a general observation of the simulation, segregation is more clearly viewed as opposed to agglomeration under condition of low particle's surface energy. Snapshots on the cross section of the model granulator however showed that there are contacts between seeds and fine particles surrounding them thus, this proves the potential seeded granules formation. Simulations were also carried out for different set of parameters, by manipulating drum rotational speed and seeds/fine size ratio to see which conditions give rise to formation of seeded granules.

It should be noted that for the results in these variable studies, the seeded granules are taken as those which have a seed contacting with fine particles, regardless of its surface coverage. Results showed that higher numbers of potential seeded granules can be produced at low drum rotational speed, low particle's surface energy and high seed/fines size ratio.

5.1 Future Work Recommendation

Further refinement of these results needs to be performed to know percentage coverage of fines for each seeds. Investigation could also be planned to study the effect of binder viscosity over the mass of particles, despite over the particle's area as what has been studied in this work. Each particle's movement from the start to the end of simulation should also be tracked down for more dependable and detailed results.

REFERENCES

- Betz, B., Junker-Buzgin, P., Leunberger, H., 2003. Batch and continuous processing in the production of pharmaceutical granules. *Pharmaceutical Development and Technology*, 8 (23), 289-297
- Contact Mechanics Lectures, 2012, retrieved from http://courses.washington.edu/overney/ChemE554_Course_Mat/course_material/Ch_2_Contact%20Mechanics.pdf
- Cundall, P.A., Strack, O.D.L., 1979. A discrete numerical method for granular assemblies. *Geotechnique* 29 (1), 47-65
- Deen, N.G., Van Sint Annaland, M., Van der Hoef, M.A., Kuipers, J.A.M., 2007. Review of discrete particle modeling of fluidized beds. *Chemical Engineering Science* 62, 28-44
- Hassanpour, A., Kwan, C.C., Ng, B.H., Rahmanian, N., Ding, Y., Anthony, S.J., Jia, X.D., Ghadiri, M., 2008. Effect of granulation scale-up on the strength of granules. *Powder Technology* 189 (2009) 304-312
- Hassanpour, A., Susana, L., Pasha, M., Santomaso, A. C., Ghadiri, M., 2011. Discrete Element Modelling of Seeded Granulation in High Shear Granulators. *Effect of granulation scale-up on the strength of granules. Powder Technology* 189 (2009) 304-312
- Hertz, H., 1882. Uber die Berührung fester elastischer Körper. *Journal für die Reine und Angewandte Mathematik* 92, 156-171.
- Johnson K, L., Kendall, K., Roberts, A. D., 1971. Surface Energy and the Contact of Elastic Solids, *Proc. R. Soc. London*, A324, 301-313.
- Mindlin, R.D., Deresiewicz, H., 1953. Elastic spheres in contact under varying oblique forces. *Journal of Applied Mechanics- Transactions of the ASME, Series E* 20, 327-344
- Rahmanian, N., Ng, B.H., Hassanpour, A., Ghadiri, M., Ding, Y., Jia, X., Anthony, J., 2008. Scale-up of high shear mixer granulators. *KONA* 26, 190-204
- Rahmanian, N., Ghadiri, M., Jia, X., 2011. Seeded Granulation. *Powder Technology* 206 (2011) 53-62

Tousey, M.D., 2002. The Granulation Process 101: Basic Technologies for Tablet Making. *Pharmaceutical Technology, Tableting and Granulation*, 1-13

Tsuji, Y., Tanaka, T., Ishida, T., 1992. Lagrangian numerical simulation of plug flow of cohesionless particles in a horizontal pipe. *Powder Technology* 71, 239-250

Vervaet, C., Remon, J.P., 2005. Continuous granulation in the pharmaceutical industry. *Chemical Engineering Science* 60 (2005) 3949-3957

Zhu, H.P., Zhou, Z.Y., Yang, R.Y., Yu, A.B., 2007. Discrete particle simulation of particulate systems: Theoretical developments. *Chemical Engineering Science* 62 (2007) 3378-3396

Zhu, H.P., Zhou, Z.Y., Yang, R.Y., Yu, A.B., 2008. Discrete particle simulation of particulate systems: A review of major applications and findings. *Chemical Engineering Science* 63 (2008) 5728-5770