ISSN: 2354-5917

PROCEEDINGS of

INTERNATIONAL SEMINAR ON CHEMICAL ENGINEERING

IN CONJUNCTION WITH

SEMINAR TEKNIK KIMIA SOEHADI REKSOWARDOJO (STKSR) 2017

Clean-Sustainable Process and Product Technology

2-3rd October 2017
East Hall and West Hall
Institut Teknologi Bandung

Organized by:

Sponsored by:

PT PETROKIMIA GRESIK

Supported by:

IATK-ITB
Ikatan Alumni Teknik Kimia
Institut Teknologi Bandung

PROCEEDING

International Seminar on Chemical Engineering
in conjunction with
Seminar Teknik Kimia Soehadi Reksowardojo
(STKSR) 2017

"Clean-Sustainable Process and Product Technology"

ISSN: 2354-5917

2nd-3rd October 2017
West and East Hall
Institut Teknologi Bandung
Jl. Ganesha 10 Bandung INDONESIA

Organized by

Department of Chemical Engineering
Faculty of Industrial Technology
Institut Teknologi Bandung

INTERNATIONAL SCIENTIFIC COMMITTEE

Prof. Johan Sanders. Wageningen University, the Netherlands
Prof. Ir. Dwiwahju Sasongko, M.Sc., Ph.D
Prof. Dr. Eng. I Made Joni, M.Sc
Assoc. Prof. Dr. M. Akbar Rhamdani, Ph.D
Assoc. Prof. Dr. Oki Muraza

STEERING COMMITTEE

Prof. Dr. Herri Susanto. Institut Teknologi Bandung
Prof. Dr. Subagjo Institut Teknologi Bandung
Prof. Dr. Ir. Mubiar Purwasasmita Institut Teknologi Bandung
Prof. Tjandra Setiadi, Ph.D. Insitut Teknologi Bandung
Prof. Dr. Yazid Bindar. Institut Teknologi Bandung
Prof. Johnner Sitompul, Ph.D. Insitut Teknologi Bandung
Prof. I. G. Wenten, Ph.D Insitut Teknologi Bandung
Assoc. Prof. Tatang Hernas Soerawidjaja. Institut Teknologi Bandung

ORGANIZING COMMITTEE

Chairman: Hary Devianto, Ph.D.

Dr. Pramujo Widiatmoko
Ardiyan Harimawan, Ph.D.
M.T.A.P. Kresnowati, Ph.D.
Dr. Antonius Indarto
Dianika Lestari, Ph.D.
Dr. Jenny Rizkiana
Dr. Vita Wonoputri
Dr. Dian Shofinita
Meiti Pratiwi, M.T.
Dr. Anggit R.

Agnes Veronica Victoria
Devina Regina
Catharine Bella
Agatha Victoria
Vania Elita Krisnandika
Nicholas George Stanley
The Arif Setio Nugroho
Catherine Melinda
Bonar Mangara Trijaya Manurung
Ghiffary Azka Nur Aulia
Judistira
Faisal Anggi Rangkuti
James Wijaya
Vincentius Ferry
Budi Prakarsa
Yeremia William Susanto
Kreszen Livianus Gatalie

ISSN: 2354-5917

MESSAGE

Welcome to all honorable keynote speakers, presenters, and participants in the International Seminar on Chemical Engineering in conjunction with Seminar Teknik Kimia - Soehadi Reksowardojo (STKSR) 2017. This annual seminar has been an event of research dissemination and knowledge sharing for chemical engineering community in Indonesian, as well as neighboring countries.

Chemical engineering concern to produce, transform, transport, and properly use of chemical, material, and energy. This field of engineering is therefore closely related to industry development in a country. Process design and analysis of technology, chemical reaction engineering, and safety analysis are the main object of chemical engineering. Chemical engineering has also significant role to fulfill one of millennium development goals, i.e. to ensure environmental sustainability. With limit on resources and increasing environment effect due to industries, we need to move toward a clean-sustainable processing technology.

We encourage youth and motivated professional to develop and share their innovation in the clean-sustainable product and process technology through this seminar. Hopefully, the seminar will bring a productive and fruitful discussion. Lastly, we greatly thanks all sponsors and contributors to support this seminar.

Prof.Dr.Ir. Kadarsah Suryadi, DEA.

Rector of Institut Teknologi Bandung (ITB)

ISSN: 2354-5917

MESSAGE

Dear Colleagues,

On behalf of the Organizing Committee of the International Seminar on Chemical Engineering, I am gladly welcome you all to Institut Teknologi Bandung, Bandung, Indonesia. This year, the Seminar which is held in conjunction with Seminar Teknik Kimia - Soehadi Reksowardojo (STKSR) 2017 raises the topic of 'Clean-sustainable Process and Product Technology'. Through this topic, we are promoting an awarness on global warming issues and sustainable production.

Those topics will be addressed in several categories, i.e. bionergy, chemurgy, alternative energy, fossil energy and mineral processing, process technology, and advance science in plenary lectures and parallel sessions. Here, we encourage young and inspiring keynote speakers to share their energy and experiencies. In end of seminar, we invite speakers in a workshop session who will share their industrial/field experiences.

We have prepared social (get-together) event, so that delegates may meet and communicate one another. Finally, the committee gratefully addressed all sponsors and ChemEng-ITB Alumni for funding and supports. We also thank all International/Technical Committee members, all the plenary and invited speakers and all oral/poster presenters for their kind efforts and contributions in making this conference a success.

Thank you

Hary Devianto, Ph.D.

Chairman of STKSR 2017

ISSN: 2354-5917

ACKNOWLEDGEMENT

The completion of this undertaking could not have been possible without the participation and assistance of so many people whose names may not all be enumerated. The contributions are sincerely appreciated and gratefully acknowledged. However, we would like to express our especially deep appreciation and gratitude to the following.

1. Institut Teknologi Bandung, Faculty of Industrial Technology, Department of Chemical Engineering.
2. Lembaga Penelitian dan Pengabdian kepada Masyarakat Institut Teknologi Bandung (LPPM ITB)
3. PT. Petrokimia Gresik
4. Medco Foundation
5. Ir. Rauf Purnama, IPU
6. Biomass Gasification Research Group Department of Chemical Engineering Institut Teknologi Bandung

for their support toward STKSR 2017. We sincerely hope that our good cooperation can extend to other opportunities in the future.

ISSN: 2354-5917

PG

PT PETROKIMIA GRESIK

TABLE OF CONTENT

ISSN: 2354-5917

POSTER PRESENTATION SESSION

ISSN: 2354-5917

Granulation of NPK Fertilizers

I Dewa Gede Arsa Putrawan[1,*], Dendy Adityawarman[2],
Haris Askari[3], and Natal Bagus Jalu Buana[3]

[1]Research Group on Chemical Product Design and Development,
[2]Research Group on Chemical Process Design and Development,
[3]Study Program of Chemical Engineering,
Faculty of Industrial Technology, Institut Teknologi Bandung
Jalan Ganesha 10, Bandung 40132, Indonesia

** Corresponding Author's E-mail: idewa@che.itb.ac.id*

Abstract
The purpose of this research is to study the physical granulation of mixed NPK fertilizers consisting of urea, diammonium phosphate, and potassium chloride. In addition, eggshell was used as filler. The granulation was carried out in a pan granulator with a pan diameter of 40 cm at a rotation speed of 35 rpm and an inclination angle of 45°. Aqueous molasses and water were used as binding liquid. The granulation variations included molasses concentration, location of liquid spray, and liquid-to-solid (LS) ratio. The granulation performance was evaluated by measuring the yield and compressive strength of onsize fraction (2-4 mm). Molasses concentration and LS ratio increased the onsize fraction. The optimum onsize fraction using hot water and 50%-molasses solution as binding liquid were 30% and 40%, respectively. Hot water resulted in higher compressive strength than 50%-molasses. Better granulation was found when the location of liquid sprayer was divided into two sections, on the bulk flows near scrapper and on the rolling regions of powder flow.

1. Introduction

NPK fertilizers are multinutrient fertilizers which contain all macronutrients needed by the plants, i.e., nitrogen (N), phosphor (P), and potash (K). Bulk production of NPK fertilizers are mainly in the granular form, with the advantage of having excellent storage, handling, and transport properties. Granular NPK fertilizers may be prepared by reactive granulation or physical granulation. In the former process, phosphoric acid is made to react with ammonia vapor to produce ammonium phosphate slurry. The slurry is then sprayed upon a tumbling bed of potassium chloride, recycled solid, filler and other solid materials to form NPK granules. In the later, mixed fertilizers are tumbled with the addition of binder. These two processes are sometimes called as slurry and solid routes, respectively[1].

Fillers are used to adjust the percentage of the nutrients to a desired ratio, prevent caking of the fertilizers, and increase the fertilizer weight[2]. Fillers dilute the concentration of fertilizer active ingredients, which can burn delicate roots and stems. Fillers contribute a significant portion in NPK formulation. NPK 20-10-10 granulated from urea, DAP (diammonium phosphate), and KCl, for example, contains 36% fillers by weight. Fillers are usually taken from inorganic minerals, such as zeolite, clay, dolomite, phosphate rock, and bentonite. These materials are not renewable and their reserves are, of course, limited. Alternatives for conventional filler materials are required.

In seeking alternatives for materials, attentions have to be paid on agricultural and livestock wastes. Eggshell can be considered as an alternative for NPK fillers. Fig. 6 shows the estimated volume of eggshell production which was estimated based on the world production of eggs from FAO[3] and assuming that an average 11% weight of eggshell in egg[4]. As seen from the figure, the world production of eggshell reached 7 million tonnes per year.

Eggshell is a bio-ceramic composite material with an extracellularly assembled structure, whose function is to protect the contents of the egg and to ensure the calcium necessary for the formation of the chick's skeleton. Eggshells are comprised of a network of protein fibers, associated with crystals of calcium carbonate (96% of shell weight), magnesium carbonate (1%) and calcium phosphate (1%), and also of organic substances and water. Calcium carbonate ($CaCO_3$), the major constituent of the shell, is an amorphous crystal that occurs naturally in the form of calcite (hexagonal crystal)[5]. Although many works on NPK granulation have been published[6-10], intensive study on the granulation of NPK fertilizer with eggshell as filler is not available.

This research is aimed to study the physical granulation of NPK fertilizer with eggshells as fillers. In more specific, this research was proposed to study the particle size distribution and compressive strength of granules obtained from physical granulation of NPK fertilizers with eggshell as filler. Physical granulation was selected instead of reactive granulation as physical granulation requires lower capital cost and could be handled by home industries. Urea, DAP, and KCl were used as the nitrogen, phosphor, and potassium sources. Molasses and hot water were used as binder. At first, the flow behaviors of single materials during the granulation were observed. Secondly, the granulations of mixed fertilizer were studied by observing the particle size of the granulated products. The compressive strengths of onsize granules were then measured and compared to those of granules using bentonite, a representative of conventional fillers.

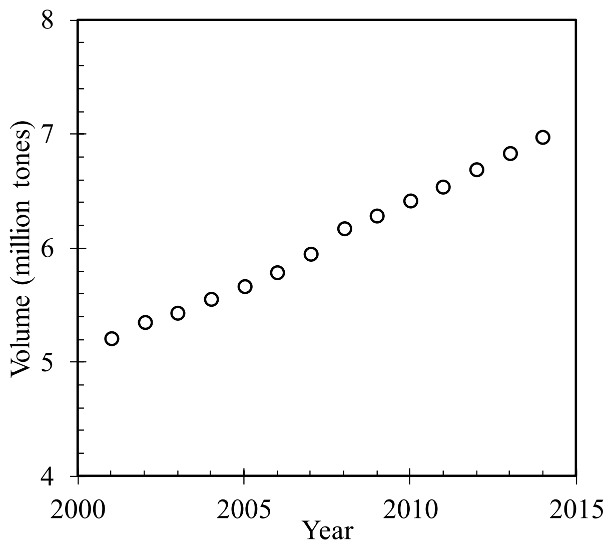

Fig. 6. World production of eggshell.

2. Experiments

2.1 Materials

Urea and KCl were purchased from market, having NPK analysis of 46-0-0 and 0-0-60, respectively. DAP having an analysis of 15-46-0 was kindly supplied by PT. Petrokimia Gresik. Eggshell was collected from a local cake shop. Molasses was kindly supplied by PT. Madukismo, Yogyakarta. All solid materials were milled until passing 100 mesh screen before granulated.

2.2. Equipment

Fig. 7 shows the schematic of the granulation apparatus used. The apparatus consists of an inclined pan equipped with a scrapper, supported on a frame which allows slope adjustment. The pan was rotated by a motor equipped with a speed reducer and an inverter. The pan was made from flexiglass, having a diameter of 40 cm and a rim height of 4 cm. The rotation speed and inclination angle were fixed at 35 rpm and 45°, respectively.

2.3. Procedure

The granulation was carried out in batch system. About 100 g grinded materials consisting of urea, DAP, KCl, and fillers were put into the pan and then rotated. After the mixtures were tumbled homogeneously, binder was sprayed onto the tumbling materials. The granulation was stopped after a determined time. The granules were then dried in oven at 70°C and then divided into three fractions, i.e., oversize (+5 mesh or ≥ 4 mm), onsize (+5 mesh/-10 mesh or between 2 to 4 mm), and undersize (-10 mesh or ≤ 2 mm).

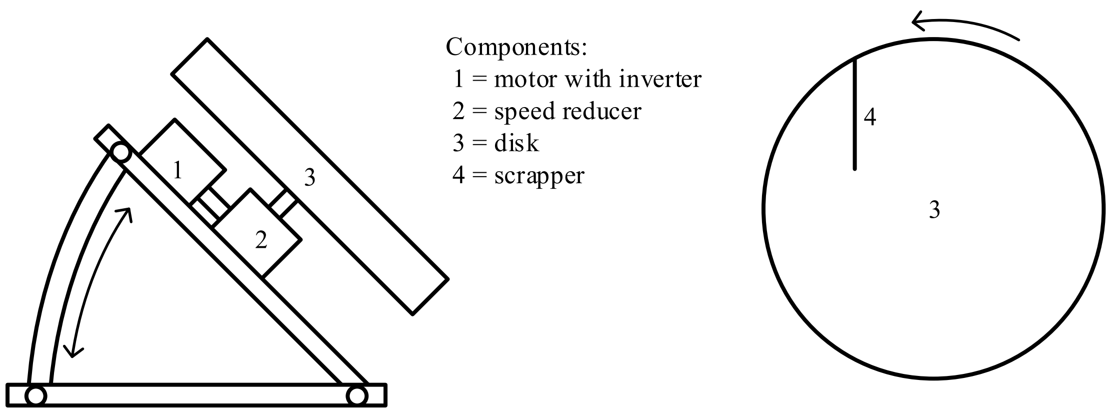

Fig. 7. Schematic of granulation apparatus.

3. Results

3.1 Flow pattern

Granulation in pan granulators are facilitated by tumbling process[11]. The homogeneity of the tumbling materials is very important when granulating solid mixtures. Studying the flow behavior of materials on a rotating pan can give description of the homogeneity of the materials when granulated. Fig. 8 presents the flow patterns arising on the pan granulator used of single materials as well as of mixed NPK and eggshell at 35 rpm. In general, flow patterns arising on a pan granulator include slipping, rolling, cascading, cataractering, and centrifuging[12]. Cascading and cataractering facilitate the consolidation of particles to collide eat other to enable growing. Rolling facilitates the colliding granules to form spherical shapes. Centrifuging is not expected because all materials are carried by centrifugal forces and adhere on the rim. Most materials used exhibited cascading, cataractering, and rolling. Eggshells which were used as fillers could be well tumbled and had almost similar pattern with bentonite. Urea, however, showed centrifuging pattern. It was caused by the low density of urea so that at the same rotation speed, urea is easily carried by centrifugal force. When eggshells were incorporated into NPK materials, consisting of urea, DAP, and KCl, the mixtures could be well tumbled. Good tumbling behavior is necessary for achieving homogeneous composition.

3.2 Effects of spray position

Spray position has important role on the extent of granulation as well as on the size distribution of granules. To study the effects of spray position, bentonite was granulated using molasses solution as binder for 5 minutes at molasses concentration of 30% and L/S ratio of 15%. There were four positions observed, i.e., at the top near the scrapper (T), at the bottom on the rolled material (B), at the middle between top and bottom (TB), and back and forth along the flowing particles between T and B (TB*). Fig. 9 shows the schematic of T, B, and TB position. The results were depicted in Fig. 10. It was found that spraying binder on the flowing material along the space from near scrapper to the rolling particles at the bottom gave the highest onsize yield (50% onsize). This spray method, however, requires that the sprayer is movable along the top and bottom.

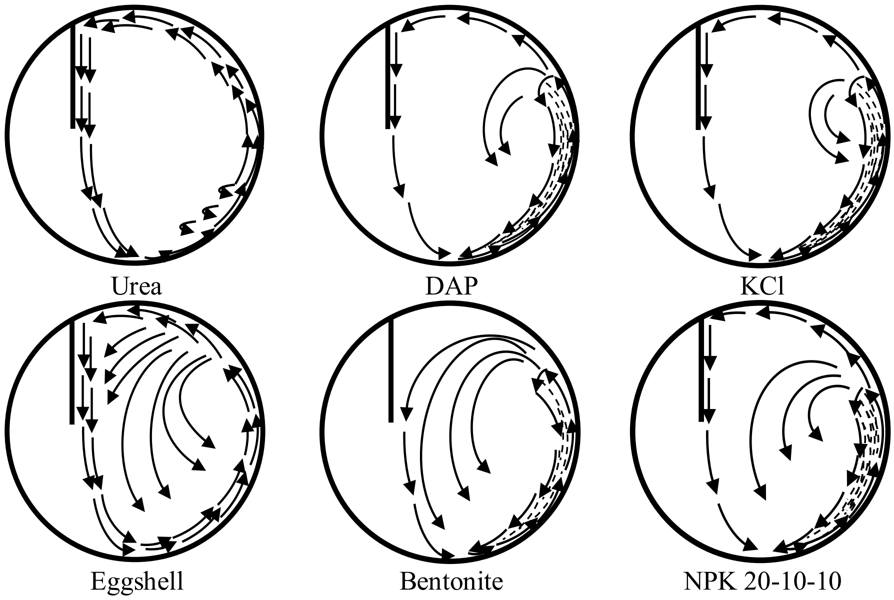

Fig. 8. Flow pattern of materials.

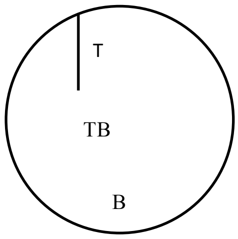

Fig. 9. Location of sprayer.

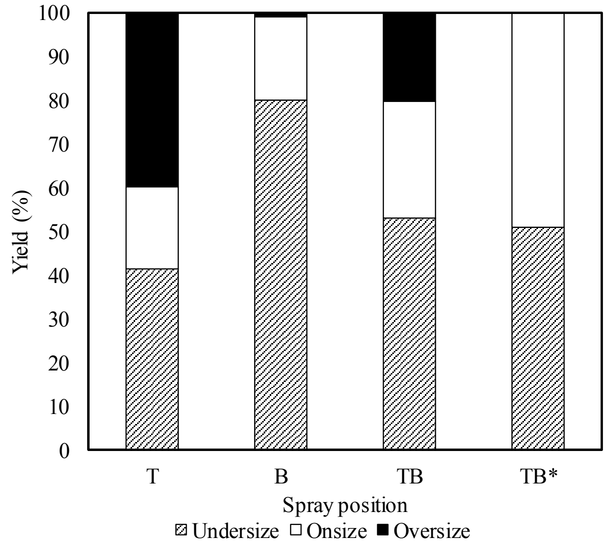

Fig. 10. Effect of sprayer position.

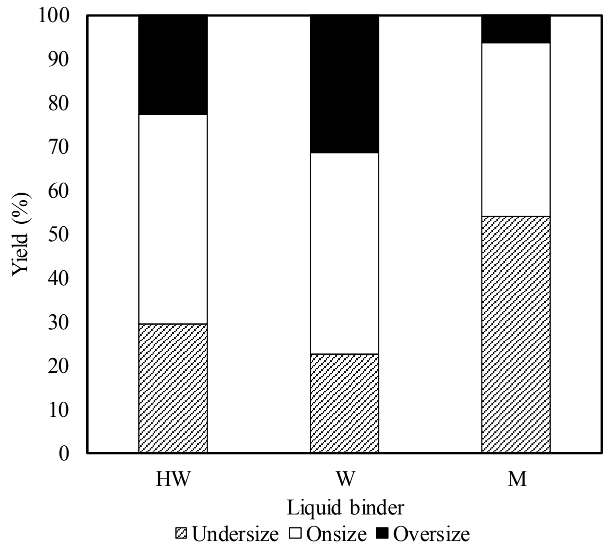

Fig. 11. Effect of binder.

3.3 Effects of binder

Three binding liquid solutions used in this research include molasses solution (M), cold water (W), and hot water (HW). The effects of liquid were studied by granulating bentonite-urea mixtures with 36% urea. This formula is corresponding to urea content of NPK 20-10-10 having high content of urea. The granulations were carried at L/S ratio of 20%. Molasses concentration used was 50%. The results are shown in Fig. 11. The order of binder in decreasing onsize yield are hot water, cold water, and molasses solution. Urea was known very soluble in water, 1079 g/L at 20°C[13]. Urea, therefore, functions not only as raw material but also as binder. The higher the temperature the higher the solubility and the faster the urea solubilizes. Molasses 50%, on the other hand, was very viscous that it is difficult to flow and to coat particles. In addition, for the same L/S ratio, it contained less water. Water without molasses, therefore, could granulate NPK particles containing urea. The role of urea as binder was convinced by granulating a bentonite-urea mixture containing 21% urea which is corresponding to NPK 15-15-15. It was found that to have the same extent of granulation to that of NPK 20-10-10, an L/S ration of 35% was necessary. It means that lower urea content requires more liquid to achieve the same onsize yield.

3.4 Effect of L/S ratio

Effect of L/S ratio was studied by using NPK 20-10-10 and with eggshell as binder. The granulations were carried for 5 minutes using hot water as binder. The results are shown in Fig. 12. As comparison, Fig. 13 shows the results for the case bentonite as filler. Eggshell as binder gave approximately the same extent of granulation to that of bentonite. It could be understood as eggshell also chemically consists of mineral having density which is close to that of bentonite. Compared to bentonite-urea mixture, L/S ratio necessary for granulating NPK is lower than that for bentonite-urea mixture. It was caused by the existence of KCl which also contributed binding properties. The solubility of KCl used is 20 g/L at room temperature. The dissolved KCl played a role as binder, which also occurred for urea. More soluble material results in stronger coalescence bridges[14]. As can be seen also that L/S ratio higher than 5% decreased onsize fraction and increased oversize fraction. It means that the L/S interval in which the granulation operates well is narrow, i.e., 0 to 5%.

The sensitivity of NPK granulation to L/S ratio could also be seen from the flow pattern of NPK during the granulation. The flow patterns are shown in Fig. 9. The particles grew fast that in four minutes, fine powders vanished and the granules could not lifted or left rolled at the bottom.

3.5 Compressive strength

Fig. 15 shows the compressive strength of onsize NPK granules obtained using hot water and 50% molasses solution as binders. The strengths were determined from 20 granules. Molasses gave lower compressive strength than hot water. The very viscous solution when molasses used as binders made the binder difficult to penetrate into the particles. Hot water, on the other hand, solubilize urea and KCl and left stronger bridge after dried. In general, 7% L/S ratio gave the highest strength. However, the granules obtained did not satisfy the required strength of minimum 3 MPa.

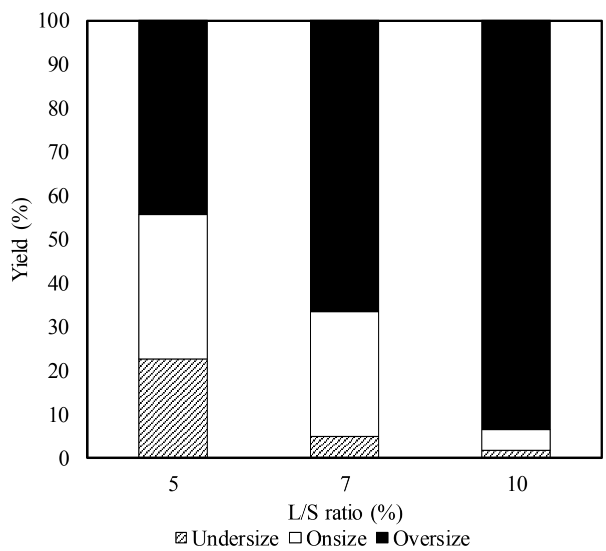

Fig. 12. Effect of L/S ratio on distribution size of NPK with eggshell as filler.

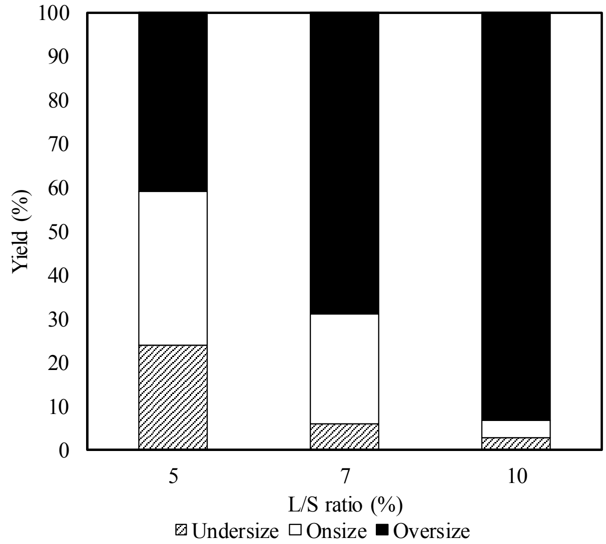

Fig. 13. Effect of L/S ratio on distribution size of NPK with bentonite as filler.

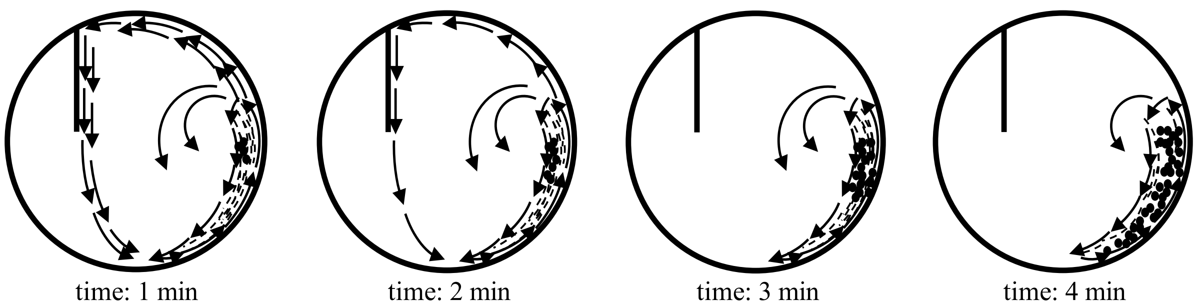

Fig. 14. Flow pattern during granulation of NPK.

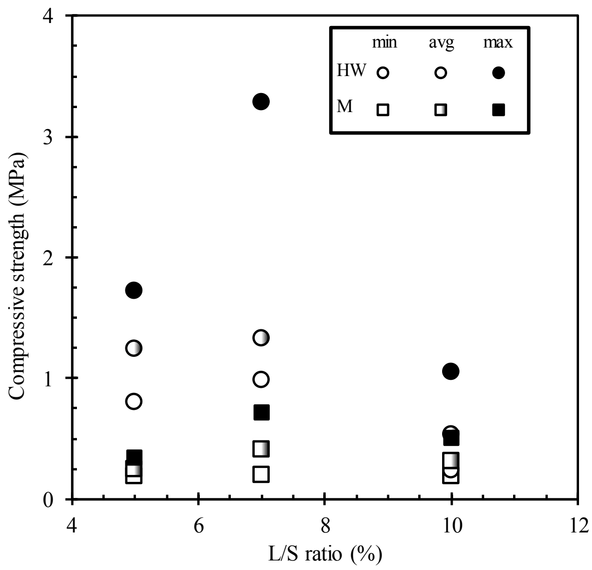

Fig. 15. Compressive strength of NPK granules.

4. Conclusions

NPK mixers consisting urea, DAP, and KCl have been granulated using a pan granulator and eggshell as filler. The granulation performance was evaluated by measuring the yield and compressive strength of onsize fraction (2-4 mm). The granulation using eggshell as filler had the same granulation extent to that using bentonite as filler. Hot water was found to be better binding liquid than cold water and molasses solution for facilitating the collisions of particles. Molasses concentration and L/S ratio increased the onsize fraction. The optimum onsize fraction using hot water and 50%-molasses solution as binding liquid were 30% and 40%, respectively. The yield of onsize fraction was significantly affected by liquid-to-solid ratio. Hot water resulted in higher compressive strength than 50%-molasses. Better granulation was found when the location of liquid sprayer was divided into two sections, on the bulk and on the rolling regions of powder.

Acknowledgements

We express our thanks to PT. Petrokimia Gresik for providing DAP and to PT. Madukismo for providing molasses. This research was supported by Institut Teknologi Bandung under the grant of P3MI 2017.

References

1. Hallsworth, J. A., Fortescue, W. F. Proceeding of Technical Meeting of IFA (1984).
2. Gowariker, V., Krishnamurthy, V. N., et al. The fertilizer encyclopedia. John Wiley, New Jersey (2009).
3. FAO. Livestock statistic report. http://faostat.fao.org (2017).
4. Zaheer, K. An Updated Review on Chicken Eggs: Production, Consumption, Management Aspects and Nutritional Benefits to Human Health. *Food and Nutrition Science*; **6**: 1208-1220 (2015).
5. Oliveira, D. A., Benelli, P., et al. A literature Review on Adding Value to Solid Residues: Egg Shells. *Journal of Cleaner Production*; **46**: 42-47 (2013).
6. Olive, B. M., Hardesty, J. O. Fertilizer Granulation, Effect of Particle Size of Potassium Chloride on Granulation of Mixed Fertilizer. *Agricultural and Food Chemistry*; **12**(6), 492-497 (1964).
7. Walker, G. M., Holland, C. R, et al. Drum granulation of NPK fertilizers. *Powder Technology*;107:282-288 (2000).
8. Walker, G. M., Holland, C. R., et al. Prediction of Fertilizer Granulation: Effect of Binder Viscosity on Random Coalescence Model. *Industrial and Engineering Chemistry Research*; **40**: 2128-2133 (2001).
9. Chudinova, O. A., Poilov, V. Z., et al. Physicochemical Foundations and Optical Parameters of the Granulation Process of NPK Fertilizers based on Ammonium Sulfate. *Russian Journal of Applied Chemistry*; **85**: 933-940 (2010).
10. Farahani, S. S., Rajabipour, A., et al. Energy Use and Economic Analysis of NPK-15:8:15 Fertilizer Granulation Process in Iran. *Journal of Saudi Society of Agricultural Sciences*; **16**:265-269 (2017).
11. Sherrington, P. J., Oliver, R. Granulation. Heyden&Son, London (1981).
12. Chadwick, P.C., Bridgwater, J., Solids Flow in Dish Granulators. *Chemical Engineering Science*; **52**: 2497-2509 (1997).
13. Green, D. W., Perry, R. H. Perry's Chemical Engineers' Handbook. McGraw-Hill, New York (2008).
14. A. A. Adetayo, J. D. Litster, M. Desai, The Effects of Process Parameters on Drum Granulation of Fertilizers with Broad Size Distributions. *Chemical Engineering Science*; 48: 395l-3961 (1993).