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Department of Chemical Engineering, National Taiwan University of Science and Technology, 43 Keelung Road, Section 4, Taipei 106-07, Taiwan

Renita Manurung, Rakhmat Akbar Sinaga and Rahmad Taufik Simatupang
Department of Chemical Engineering, University of Sumatera Utara, Medan 20155 Indonesia

R&D Center for Membrane Technology and Department of Chemical Engineering, Chung Yuan Christian University, Chung-Li 32023, Taiwan, R.O.C.

Department of Chemical Engineering, Hanoi University of Technology (HUST), No. 1 Dai Co Viet Str., Hanoi, Vietnam.
Department of Quality Management, HUST, No. 1 Dai Co Viet Str., Hanoi, Vietnam.

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Erwan Adi Saputro, Kusno Budikardjono, Ali Altway
Chemical Engineering Department, UPN Veteran Jawa Timur, Surabaya, Indonesia
Chemical Engineering Department, ITS Surabaya Indonesia.

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Nanako Hagihara, Mitsuru Sasaki, Armando T. Quitain, Takuma Higashiura, Motonobu Goto
Graduate School of Science and Technology, Kumamoto University 2-39-1 Kurokami, Chuo-ku, Kumamoto 860-8555 Japan
Research Institute, KAGOME CO., LTD.
17 Nishitomiyama, Nasushiobarashi, Tochigi 329-2762 Japan
Department of Chemical Engineering, Nagoya University Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

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Junel B. Borbo and Mark Daniel G. de Luna
Department of Chemical Engineering, University of the Philippines Dilimanand Departmentof Chemical Engineering, Bicol University
Department of Chemical Engineering, University of the Philippines Diliman

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Mohammad Jakir Hossain Khan, M. A. Hussain
Department of Chemical Engineering, Faculty of Engineering, University of Malaya, 50603, Kuala Lumpur, Malaysia

Department of Chemical Engineering, Sepuluh Nopember Institute of Technology, Surabaya 60111, Indonesia

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Norfarhana Ab. Samad, Nazwa Jon, Rizafizah Othaman and Ibrahim Abdullah
School of Chemical Sciences and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi Selangor, Malaysia.

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Chantamanee Poonjarernsilpa, Noriaki Sano, Taiga Ishii, and Hajime Tamon
Department of Chemical Engineering, Graduate School of Chemical Engineering Kyoto University, Kyoto 615-8510, Japan
Department of Chemical Engineering, Faculty of Engineering, Rajamangala University of Technology Krungthep, 2 Nanglinchee road, Sathorn, Bangkok 10120, Thailand

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MariaChristinaPrihatiningsih,ImamPrasetyo, Rochmadi
DepartmentofNuclearChemicalEngineering
PolytechnicInstituteofNuclearTechnology–NationalNuclearEnergyAgency, Yogyakarta55281,Indonesia
DepartmentofChemicalEngineering
GadjahMadaUniversity,Yogyakarta55281,Indonesia

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Rodel D. Guerreroa, Emmanuel P. Belostrino, Mark Louis H. Lagura, Billy Joe Y. Uy, Terence P. Tumolva and Masatoshi Kubuochi
Department of Chemical Engineering, University of the Philippines, Diliman 1101 Quezon City, Philippines TELEFAX: +6329296640
Department of Chemical Engineering, Tokyo Institute of Technology, 2-12-1 O-Okayama Meguro-ku, Tokyo, 152-8552 Japan
Ceramics Engineering/Chemical Engineering/Metallurgical Engineering Department, Mindanao State University-Iligan Institute of Technology, Iligan City, Lanao del Norte, Philippines

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IDG. Arsa Putrawan and H. Mohamed
Research Group on Chemical Engineering Product Design and Development Faculty of Industrial Technology Insitut Teknologi Bandung, Jalan Ganesha 10, Bandung 40132, Indonesia

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Noridah B. Osman, Armando G. McDonald, and Marie-Pierre G. Laborie
Center for Biofuel and Biochemical Research, Universiti Teknologi PETRONAS, Perak 31750, Malaysia
Renewable Materials Program, Department of Forest, Range and Fire Sciences, University of Idaho, USA
Institute of Forest Utilization and Work Sciences, University of Freiburg, Germany

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Department of Materials and Metallurgical Engineering
Sepuluh Nopember Institute of Technology, Surabaya60111Indonesia
Department of Mechanical Engineering, Surabaya State Shipbuilding Polytechnic, Sepuluh Nopember Institute of Technology (ITS), Surabaya 60111, Indonesia
Department of Materials Science and Engineering, National Taiwan University of Science and Technology (NTUST), Taipei, Taiwan
Department of Electronic Engineering, National Taiwan University of Science and Technology (NTUST), Taipei, Taiwan

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Universiti Kebangsaan Malaysia
Universiti Tun Hussein Onn Malaysia

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Department of Chemical Engineering, King Saud University, Riyadh, Saudi Arabia
SABIC Polymer Research Center, King Saud University, Riyadh, Saudi Arabia
Research and Development Department, Printpack Inc., Williamsburg, USA

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Polymer Engineering Laboratory, Department of Chemical Engineering
Faculty of Engineering, Chulalongkorn University, Bangkok 10330, Thailand

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Anucha Pengdam and Sarawut Rimdusit

Polymer Engineering Laboratory, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University, Payathai Road. Pathumwan, Bangkok, 10330, THAILAND.

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Hajime Tamo, Takuya Akatsuk, Hiroki Mori, and Noriaki Sano
Department of Chemical Engineering, Kyoto University, Katsura, Kyoto 615-8510, Japan

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Ahmad Shamiri, M. A. Hussain, Farouq Sabri Mjalli, Navid Mostoufi
Department of Chemical Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia
Training center, Razi Petrochemical Company, P.O. Box 161, Bandar Imam, Iran
Petroleum & Chemical Engineering Department, Sultan Qaboos University, Muscat, 123, Oman
Process Design and Simulation Research Center, School of Chemical Engineering, College of Engineering, University of Tehran, P.O. Box 11365/4563, Tehran, Iran

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Department of Mechanical and Systems Engineering,University of Hyogo, 2167 ShoshaHimeji 671-2280Japan

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Hyung Woo Lee, Johnner P. Sitompul, and Yeung Ho Park
Department of Chemical Engineering, Faculty of Industrial Technology, Institute of Technology Bandung, Jl. Ganesha 10, Bandung 40132, Indonesia
Department of Materials and Chemical Engineering,Hanyang University, Ansan,Gyeonggi-do 426-791, South Korea

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Department of Chemical Engineering, Chulalongkorn University, Bangkok 10330 Thailand
Department of Chemical Engineering, Khon-Kaen University, Khon-Kaen 40000 Thailand

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Department of Chemical Engineering, Faculty of Engineering and Industrial Technology Silpakorn University, Nakhon Pathom 73000, Thailand

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Dien Nurfathi, Ulfa Hardyanti, Agus Purwanto
Department of Chemical Engineering, Sebelas Maret University, Surakarta632112, Indonesia

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Department of Chemical Engineering HoChiMinh City University of Technology, VNU-HCM, Vietnam

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Energy Engineering Program, College of Engineering University of the Philippines Diliman, Quezon City 1101 Philippines
Department of Chemical Engineering University of the Philippines Diliman, Quezon City 1101 Philippines
Insitute of Chemistry University of the Philippines Diliman, Quezon City 1101 Philippines
National Sciences Research Institute University of the Philippines Diliman, Quezon City 1101 Philippines

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Department of Chemical Engineering, Faculty of Engineering, Prince of Songkla University, Hat Yai, Songkhla 90112

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Jirawat Kajohnchaiyagua, Chanchira Jubsilp, and Sarawut Rimdusit
Polymer Laboratory Engineering, Department of Chemical Engineering, Faculty of Engineering,Chulalongkorn University, Payathai, Pathumwan, Bangkok, 10330, THAILAND.
Department of Chemical Engineering, Faculty of Engineering, Srinakharinwirot University, Nakhonnayok 26120, THAILAND

Department of Chemical Engineering, Sepuluh Nopember Institute of Technology, Surabaya 60111, Indonesia

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Nazwa Jon , Ibrahim Abdullaha, and Rizafizah Othaman
School of Chemical Sciences and Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600, Bangi, Selangor, Malaysia

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Renung Reningtyas, Indra Perdana, I Made Bendiyasa
Department of Chemical Engineering, Faculty of Engineering Gadjah Mada University, Jl. Grafika 2, Yogyakarta, 55281 Indonesia
Master student in Department of Chemical Engineering, Faculty of Engineering, Gadjah Mada University, Jl. Grafika 2, Yogyakarta, 55281 Indonesia

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Chemical Engineering Program, Bandung Institute of Technology, Bandung 40132, Indonesia

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Department of Chemical Engineering, University of the Philippines, Diliman, Quezon City 1101 Philippines
Department of Chemical Engineering, Tokyo Institute of Technology, Meguro-ku, Tokyo 152-8552 Japan

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Ralph P. Villaa, Jonas Karl Chritopher N. Agutayaa, Terence P. Tumolvaa and Masatoshi Kubouchib
Department of Chemical Engineering, University of the Philippines, Diliman, Quezon City 1101 Philippines
Department of Chemical Engineering, Tokyo Institute of Technology, Meguro-ku, Tokyo 152-8552 Japan

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Masao Gena, Hideo Kakutac, Yoshihito Kamimotod and Wuled Lenggoroa
Graduate School of Bio-Applications and Systems Engineering, Department of Chemical Engineering and Institute of Engineering, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184-8588, Japan
Plant Ecochemicals Research Center, Eniwa, Hokkaido 061-1374, Japan Kanagawa Industrial Technology Center, Ebina, Kanagawa 243-0435, Japan

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Bidhari Pidhatikaa, Mathias Rodenstein, Yin Chena, Marcus Textora, and Rupert Konradia
Laboratory for Surface Science and Technology, Department of Materials, ETH Zürich, Switzerland
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Department of Chemical Engineering, Curtin University, Perth, GPO Box U 1987, 6845, Western Australia-Australia
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University of Wollongong, NSW 2522, Australia.
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Agus Purwanto, and HendriWidiyandari
Department of Chemical Engineering,SebelasMaret University,Jl. Ir. Sutami No. 36 A, Surakarta, Indonesia
Department of Physics,Diponegoro University,Jl. Prof. H. Soedarto SH, Semarang, Central Java 50275, Indonesia

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Agung Nugroho, Widiyastuti, and Sugeng Winardi
Department of Health and Safety Engineering, Surabaya Shipbuilding State Polytechnic, 60111, Indonesia
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Department of Materials Engineering, Akita National College of Technology, Akita 010-8511 Japan

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S.Muryantoa, A.P.Bayuseno, and E.Supriyo
Dept. of Chemical Engineering and Office of Research, UNTAG University in Semarang, Bendhan Dhuwur Campus, Semarang 50192, Indonesia
Centre for Waste Management, Mechanical Engineering Graduate Program, Diponegoro University, Tembalang Campus, Semarang 50275, Indonesia
Chemical Engineering Vocational-D3 Program, Diponegoro University, Tembalang Campus, Semarang 50275, Indonesia

Department of Chemical Engineering, University of the Philippines, Diliman, Quezon City, Philippines

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Kristine Marfe S. Amer, Maria Lourdes P. Dalid, PhD, and Ming-Chun Lu, PhD
Environmental Engineering Program, University of the Philippines Diliman, Quezon City 1101 Philippines
Department of Chemical Engineering, University of the Philippines Diliman, Quezon City 1101 Philippines
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Department of International Development Engineering, Tokyo Institute of Technology, Tokyo 152-8550 Japan

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Hadiyantoand Danny Soetrisnanto
Center of Biomass and Renewable Energy (CBIORE)
Chemical Engineering Department, Diponegoro University
Jln. Prof. Sudharto, Tembalang, Semarang, 50239, Telp/Fax: (024)7460058

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Aries A. Arcega, Chi-Chuan Kan, Maria Lourdes P. Dalida, Meng-Wei Wan
Department of Chemical Engineering, University of the Philippines Diliman, Quezon City 1101,Philippines
Department of Environmental Engineering and Science, Chia Nan University of Pharmacy andScience, 60, Erh-Jen RD., Sec.1, Jen-Te, 717, Tainan, Taiwan

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Zilfahmiati, Ronny Purwadi
Department of Chemical Engineering – Faculty of Industrial Technology, Institut Teknologi Bandung

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Yoshinori YAMADA, Xiaosong SUN, and Mikio SAKAI
Department of Systems Innovation, Graduate School of Engineering, University of Tokyo
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Center of Excellence on Catalytic Reaction Engineering, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University, Bangkok, 10330, Thailand

Ad-4 Synthesis of Gold Nanostructures Using Paper for Active SERS Substrate
Yian Tai, Sudeshna Kar, and Christa Desmonda
Department of Chemical Engineering, National Taiwan University of Science and Technology, Taipei 10607 Taiwan

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Granulation of Organic and Inorganic Mixtures

IDG. Arsa Putrawan* and H. Mohamed

Research Group on Chemical Engineering Product Design and Development
Faculty of Industrial Technology
Institut Teknologi Bandung, Jalan Ganesha 10, Bandung 40132, Indonesia

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

Abstract

This research was aimed to study the crushing strength of granular fillers from organic (compost) and inorganic (zeolite and clay) mixtures. Preliminary study using molasses solution as binder showed that the strength of granules obtained were unsatisfied due to the very low water solubilities of the raw materials. Urea, which is water soluble, was then added as raw material. A two-level full factorial design was carried out on small dish to study the factors which supposed to influence in the crushing strength of granules. The experimental results showed that for size range of 2 to 4 mm, the average crushing strength was found in the range of 0.015 to 1.143 kg. Urea was found to have significant effect on crushing strength. To improve the economic aspect without reducing the crushing strength, subsequent experiments were undertaken without urea in the solid raw material but with urea in the binder solution. Incorporating urea into the binder solution, even without urea in the solid raw material, was found to improve the crushing strength, about four times of that obtained by using molasses solution without urea. The crushing strength was not different between using manually rotated dish and automatically rotated drum.

Keywords: Granulation; Organic; Inorganic; Factorial design; Dish; Drum;

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 advantages of having excellent storage, handling, and transport properties. Granular NPK fertilizers may be prepared by granulation or blending. Granulation may be devided into reactive granulation or physical granulation. In the former granulation 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 physical granulation, mixed fertilizers are tumbled with the addition of binder. These two processes are sometimes called as slurry and solid routes, respectively, [Hallsworth and Fortescue, 1984]. Blended NPK, on the other hand, is simply formed by physical blending of N, P, K granular fertilizers and granular filler which is used to control NPK analysis.

Filler used to form blended NPK must be well granulated, similarly sized and dry to prevent segregation, caking and deterioration. Most of filler is prepared from inorganic minerals, such as zeolite, clay, dolomite, phosphate rock, and bentonite. These materials are not renewable and their reserves are, of course, limited. Organic based materials, such as compost, can be considered as a renewable alternative for granular filler production. Preparation of granular filler from organic and inorganic materials can be expected to have advantages, such as, preservation of soil, because inorganic materials are limited source, the low price of organic material will encourage investment in fertilizer sector to face the dramatically increasing in fertilizer consumption.

The purpose of this paper is to study the granulation of organic and inorganic mixtures to produce granular filler for blended NPK fertilizer. Compost and zeolite/clay are used as organic and inorganic materials, respectively. The granulation was first carried on a small dish to study the effects of raw material composition on the strength of ganules based on a full 2^4 factorial design. A typical run was then carried out in a laboratory scale rotary drum granulator for comparison.

Granulation is a particle size enlargement process. The mechanisms for granule growth include nucleation, coalescence, crushing, and layering, as shown in **Figure 1** [Sastry and Fuerstenau, 1973]. Nucleation occurs when nonparticulate matter forms new particles. Coalescence (agglomeration) is the successful collision of two particles to form single particle. The rate of coalescence may be size independent (random coalescence) or size dependent (preferential coalescence). Crushing is the abrasion of brittle particles. Layering is the addition of nonparticulate matter to the surface of particles. Coalescence, crushing, and layering are growth-death phenomena.

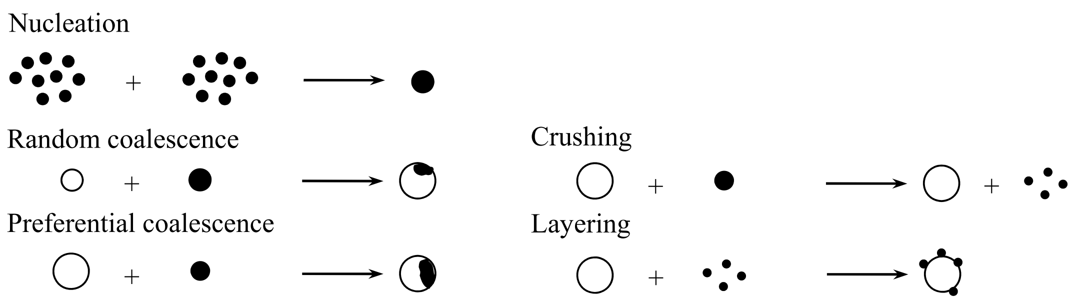

Figure 1. Mechanisms of granule formation.

Adetayo et al. [1995] have found that coalescence is the major mechanism for the granulation of diammonium phosphate, monoammmonium phosphate, and ammonium sulphate. The solution phase ratio has been identified as the governing factor in granulation, with a high ratio resulting in high degree of granulation. The solution phase ratio is defined as the ratio of volume of liquid phase to that of solid phase in the granule and is given by the following equation [Sherrington and Oliver, 1991]:

$$y = \frac{m(1+s)\rho_f}{(1-ms)\rho_l}, \tag{1}$$

where m is the binder content, s is the fertilizer solubility, ρ_f is the solid fertilizer density, and ρ_l is the liquid fertilizer density. For each granulation system, the fertilizer densities are constant and the solution phase ratio, therefore, is a function of binder content and solid solubility which is a function of granulation temperature.

2. Experimental

2.1. Materials

Zeolite and clay, which are found easily in market, were used as inorganic materials. Compost, which is commonly used for soil conditioning, was selected as organic material. In addition to these solids, urea was also involved to strengthen the granules since urea, when solubilized in water, will act as binder. Aqueous solution of molases, which is sticky enough, was used as binder solution.

2.2. Equipment

The effect of raw material compostion on granule strength was studied using a small dish granulator. 150 g of mixed solid was put inside the dish which was rotated by hand during spraying the binder solution. Binder solution was sprayed by injection with small hole to control the distribution of binder solution. Granulation time and solid-to-liquid ratio were fixed at 5 minutes and 4. Crushing strength of dry granules with size 2−4 mm was measured after drying. The experiments were carried based on a full 2^4 factorial design in which each run was conducted twice. Percentage of molasses in binder solution and the percentages of zeolite, clay, and urea, with respect to compost weight, were selected as factors. The factor levels are given below.

Table 1. Levels of factors.

Factor	Level −	Level +
Percentage of molasses in binder solution (X_1)	10	20
Percentage of zeolite from the weight of compost (X_2)	25	50
Percentage of clay from the weight of compost (X_3)	25	50
Percentage of urea from the weight of compost (X_4)	0	50

For comparison, a typical run was repeated in a rotary drum of internal diameter 250 mm and length of 250 mm. The drum is mounted on rollers connected to a variable speed motor. The drum equipped with four wedge shaped lifter bars, each 4 mm hight. **Figure 2** shows the front and side views of the granulation drum (scale in mm).

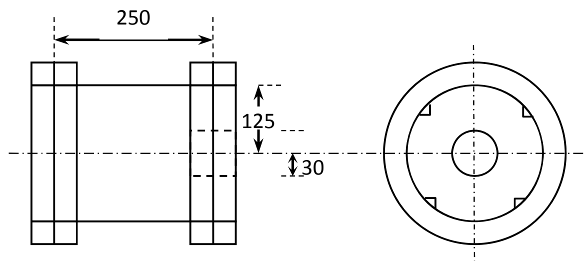

Figure 2. Schematic diagram of the granulation drum.

The drum speed was kept at 27 rpm for all experiments. This is arround 32% of critical speed which was found to be 85 rpm using commonly used equation [Sherrington and Oliver, 1991]. Binder was added into the drum by using a syringe and a stainless tube of diameter 5 mm with 1 mm drilled holes at a spacing of 5 mm. The holes on the distribution tube were arranged so that the binder was sprayed onto the tumbling granules rather than the drum wall. In preparation for an experiment, about 300 g sample of required size distribution was placed in the drum. The required amount of binder to be added was weighed and drawn into a plastic syringe. The drum was rotated for several minutes to mix the feed. While the drum was still rotating, the binder was then sprayed. The drum was rotated for the required granulation time, after which granulation is stopped and the granules were dryed.

3. Results and Discussion

The results from a small dish granulator are shown in **Table 2**. For size range of 2−4 mm, the average crushing strength of granules and the crushing strengths of individual granules were found in the range of 0.015 to 1.143 kg and 0.001 to 1.721 kg, respectively. By average, most of granules could not satisfy the required strength specification of minimum 1 kg for size range of 2−4 mm. However, compared with the granular filler prepared from compost without inorganic mineral, the crushing strength obtained has much improved. From the previous study [Tanudjaja, 2009], it was found that the maximum strength of granular filler with sizes of 2−4 mm made from compost was only 0.1 kg.

Table 2. Experimental results from dish granulation.

Run	X_1	X_2	X_3	X_4	Y_1	Y_2	Y_{ave}
1	−	−	−	−	0.019	0.045	0.032
2	−	−	−	+	0.575	0.234	0.405
3	−	−	+	−	0.034	0.036	0.035
4	−	−	+	+	0.137	0.206	0.171
5	−	+	−	−	0.075	0.031	0.053
6	−	+	−	+	0.329	0.237	0.283
7	−	+	+	−	0.037	0.015	0.026
8	−	+	+	+	0.204	0.308	0.256
9	+	−	−	−	0.062	0.081	0.072
10	+	−	−	+	0.332	0.329	0.331
11	+	−	+	−	0.039	0.042	0.040
12	+	−	+	+	0.120	0.151	0.135
13	+	+	−	−	0.052	0.058	0.055
14	+	+	−	+	0.246	0.314	0.280
15	+	+	+	−	0.032	0.046	0.039
16	+	+	+	+	0.710	1.143	0.927

The data in **Table 2** could be analyzed to obtain the effects of variables on the crushing strength. In general, the relation between response and all factors for a full 2^4 factorial design is given below:

$$Y = \beta_o + \beta_1 \cdot X_1 + \beta_2 \cdot X_2 + \beta_3 \cdot X_3 + \beta_4 \cdot X_4 + \beta_{12} \cdot X_1 \cdot X_2 + \beta_{13} \cdot X_1 \cdot X_3 + \beta_{14} \cdot X_1 \cdot X_4 + \beta_{23} \cdot X_2 \cdot X_3 + \beta_{24} \cdot X_2 \cdot X_4 + \beta_{34} \cdot X_3 \cdot X_4 + \beta_{123} \cdot X_1 \cdot X_2 \cdot X_3 + \beta_{124} \cdot X_1 \cdot X_2 \cdot X_4 + \beta_{134} \cdot X_1 \cdot X_3 \cdot X_4 + \beta_{234} \cdot X_2 \cdot X_3 \cdot X_4 + \beta_{1234} \cdot X_1\ X_2 \cdot X_3 \cdot X_4 \qquad (2)$$

where Y is response (crushing strength) and X_1, X_2, X_3, and X_4 are factors (percentages of molasses, zeolite, clay, and urea, respectively). Analysing the data shown in **Table 2** using the sandard analysis of factorial design [Montgomery, 2001], the relation between the response and the factors studied (in coded form) can be expressed as follows:

$$Y = 0.20 + 0.04 \cdot X_1 + 0{,}04 \cdot X_2 + 0.01 \cdot X_3 + 0.15 \cdot X_4 + 0.05 \cdot X_1 \cdot X_2 + 0.04 \cdot X_1 \cdot X_3 + 0.03 \cdot X_1 \cdot X_4 + 0.06 \cdot X_2 \cdot X_3 + 0.04 \cdot X_2 \cdot X_4 + 0.02 \cdot X_3 \cdot X_4 + 0.04 \cdot X_1 \cdot X_2 \cdot X_3 + 0.05 X_1 \cdot X_2 \cdot X_4 + 0.05 \cdot X_1 \cdot X_3 \cdot X_4 + 0.07 \cdot X_2 \cdot X_3 \cdot X_4 + 0.04 \cdot X_1\ X_2 \cdot X_3 \cdot X_4 \qquad (3)$$

The confidence interval (CI) for the coefficients in the above equation can be expressed by the following equation:

$$CI\ (\beta_i) = \beta_i \pm t_{\alpha/2,n-1} \cdot \sigma/\sqrt{(n)} \qquad (4)$$

where β_i is the nominal value of i^{th} coefficient, $t_{\alpha/2,n-1}$ is t value from the student's t distribution at significance level of α and degree of freedom n-1, n is number of data, and σ is standard deviation. If the limits of confidence interval of a coefficient have different signs, the effect of corresponding factor is not significant. At the confidence level of 95% and number of data of 16, the student's t value was found to be 2.13. The confidence intervals of the coefficients can be expressed as:

$$CI\ (\beta i) = \beta i \pm 0{,}04 \qquad (5)$$

Considering the confidence interval of each coefficient, it can be convinced that percentages of molasses, zeolite, and clay, have no significant effects. Only the two interaction factors between percentages of molasses and zeolite and of zeolite and clay has significant effects. The three interaction factors among percentages of molasses, zeolite, and clay has no significant effects but the effects of other three interaction factors are significant. In addition, the four interaction factors have no significant effects. Hence, the relation between crushing strength (CS) and the percentages of molasses (M), zeolite (Z), clay (C), and urea (U) can be simplified as follows:

$$CS = 0.20 + 0.15 \cdot U + 0.05\ M \cdot Z + 0.06 \cdot Z \cdot C + 0.05 \cdot M \cdot Z \cdot U + 0.05 \cdot M \cdot C \cdot U + 0.07 \cdot Z \cdot C \cdot U \qquad (6)$$

The obtained results have showed the significant effects of urea. As seen from equation (1), the solubility of solid raw material and the viscosity of binder solution [Sherrington and Oliver, 1991] have significant role to granulation degree. Urea is very soluble in water, its aqueous solubility is about 100 g/100 ml [Green, 2008]. When binder solution, which was water based binder, was sprayed onto the raw material, urea easily solubilized in the solution and resulted in viscous binder solution. This increased the thickness of the binder layer around the particles, strengthening the bridges of coalescence. The other solid materials, however, are not soluble in water that their effects on the crushing strength were not significant. In terms of granulation theory [Ennis et al., 1991], the success and fail of collision correspond to non-inertial and coating regimes of granulation, respectively. Granulation kinetic can be described in term of viscous Stoke number. Viscous Stoke number actually describes the ratio of the relative kinetic energy between colliding particles to the viscous dissipation of liquid bridge developed by the addition of binder into the surface of particles. The collision among particles will be successful when a critical viscous Stoke number is surpassed. Critical viscous Stoke number is linearly dependent on the logarithmic of the thickness of the binder layer. The increase of binder solution viscosity resulted from urea solubilization, of course, increased the thickness of the binder layer around the particles and increased the strength of ganules after drying.

In the experiment of small dish above, urea is added as raw material. The percentage of urea was also relatively high which represents 50% of the weight of organic material (compost) which may lowering the economical feasiblity of granular filler. The importance of urea in increasing the crushing strength of granules, however, has been clear. Adding urea into the binder solution was thought to be the best way to improve the economic aspect without reducing the crushing strength. Thus, further experiment was done on small dish with urea solution as binder. The experimental design was the same as run 2, except the composition binder solution. The new binder solution was composed of 20% molasses and 50% urea. In addition, the granulation with urea solution as binder in a rotaty drum granulator was also conducted for comparison. The results are given in **Table 3**.

It is clearly shown that incorporating urea into the binder solution, even without involving it in the raw material, improved the crushing strength, about four times of that obtained by using molasses solution without urea. The crushing strength, however, was not different between using manually rotated dish and automatically rotated drum. Granule obtained by drum granulator should be more densed than that obtained by small dish, since the thumbling taking place inside drum granulator gives better mixing than that generated by manually rotated dish. During granulation experiment was also observed that the extent of granulation when using small dish was higher than rotary. The high viscosity of urea containing binder solution caused a problem through spraying the binder over the solid when using drum granulator, especially for room temperature operation, as done here. The binder, henceforth, could not distribute uniformly upon the thumbling material. Small dish, with smaller quantity of material, although rotated manually, allowed binder to reach most particles during rolling the dish. This problem can be solved either by heating the sprayer or operating granulator at warm temperature.

Table 3. Results using urea solution as binder.

X_1	X_2	X_3	X_4	Y	Note
+	−	−	−	0,072	Small dish, Run 2
+	−	−	−	0,297	Small dish, with 50%-urea in binder solution
+	−	−	−	0,289	Rotary drum, with 50%-urea in binder solution

From this experiment, binder solution was found to have very significant effect on granule crushing strength in granulation of organic compost and inorganic mineral mixtures which have different surface properties resulting in relatively poor wetting. When spraying binder on the particles free spaces, liquid bridges start to appear between solid particles, then these bridges create sufficient adhesion between the particles to enable them to successfully and strongly coalesce as nuclei agglomerates. The force that holds the particles together is ultimately related to the ability of a liquid binder to wet the particles to form effective bonds. So the strength of granule depends on the strength of the individual bridge and the liquid bridge forces arise from both capillary and surface tension effects, which are static forces. However urea has great ability to wet particle and to form effective bond but the crushing strength of of most granules obtained by either rotary drum or small dish are still below 1 kg which indicate that there are many factors affecting on the wetting phenomena and not yet considered in this study.

4. Conclusion

Adding inorganic mineral into the compost could improve the crushing strength of granular filler from organic compost. Individual crushing strength 1.8 kg could be reached, although it is not desirable because it may be difficult to fragment in field. By average, however, most formulation could not achieved the specified crushing strength as there are many factors affecting the wetting phenomena and were not considered yet in this study. Crushing strength of granules obtained during granulation of organic compost and inorganic mineral on a small dish is strongly depends on the percentage of urea in the solid raw material. Analysis of a full 2^4 factoral design of experimental data from dish granulator showed that urea has very significant effecs on the crushing strength of granules. Incorporating urea into the binder solution, even without urea in the solid raw material, was found to improve the crushing strength, about four times of that obtained by using molasses solution without urea. The crushing strength, however, was not different between using manually rotated dish and automatically rotated drum. The high viscosity of urea containing binder solution was supposed to be the problem, causing the binder difficult to spray over the thumbling bed so that the binder could not distribute uniformly in the solid material.

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Nomenclature

C percentage of clay
CI confidence interval
CS crushing strength
M percentage of molasses
m binder content
n number of data

s fertilizer solubility
t t value from the student's t distribution
U percentage of urea
y solution phase ratio
Z percentage of zeolite

Greek letters
α significance level
β nominal value of coefficient
μ binder viscosity
ρ_f solid fertilizer density
ρ_g granule density
ρ_l saturated solution density
σ standard deviation

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