

Filter Cake Utilization as Filler of 15-15-15+5S Compound Fertilizer: Particle Size Distribution and Granule Crushing Strength Properties

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Abstract

Compound fertilizer which combining organic-inorganic materials need to be developed to improve the effectiveness of fertilizers in the soil. Filter cake as a material has highly potential to be used as a filler in physical process granulation of compound fertilizer. In this study, the particle size distribution and granule crushing strength properties were tested using 15-15-15 + 5S fertilizer compound formula, which are varied in the filler composition and K sources. Potassium sources consisted of 2 (two) types of fertilizers i.e KCl and K₂SO₄. Filler composition as a binder in fertilizer granulation consists of 5 combination filter cake and clay ratios (60:40, 70:30, 80:20, 90:10 and 100:0). Granulation carried out by the granulation method using pan granulator of 2 kg/batch capacity, 23 rpm rotation speed and 50° pan slope. The results of the research showed that statistically the combination of filter cake and clay 70:30 had a size distribution and hardness of granules not significantly different from standard fertilizer (100% clay).

Keywords: crushing strength; filler; filter cake; granulation; size distribution

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INTRODUCTION

Fertilizer production and fertilization technology in Indonesia are still relatively lacking, especially for compound fertilizers that combine organic-inorganic materials. Compound fertilizer which are combine organic-inorganic materials are known to be very effective in amelioration of soil,

growth and yield crops enhancement (Korai *et al.* 2014), increasing fertilizer efficiency, decreasing phosphat fixation (Utami *et al.* 2012, De Moraes *et al.* 2018), and increasing N, P, Ca and cation exchange capacity (CEC) (Ayeni *et al.* 2017, Yao *et al.* 2018;).

Granular compound fertilizers can be produced by physical or chemical granulation process. Chemical

granulation process creates homogeneous compound fertilizer however need relatively high and complex technology and huge investment. In another site, with a simpler process, physical granulation also can produce homogeneous compound fertilizer. The difference between the two methods is that the physical granulation process requires binders which are generally clay material. According to IFDC (1998) the weakness of clay material as a binder that clay has a large water holding capacity, which is difficult for the drying process and contains very little nutrients.

The quality of granule compound fertilizer from the wet granulation process can be assessed from the distribution of granule size and hardness. There are three groups of size distribution of compound fertilizer granules: 1) under size: particle size under standard product size (<2 mm), 2) On size: 2-4 mm particle size, and 3) Over size: particle size greater than standard product size (>4 mm). The amount of fertilizer granules below or above the standard size will be returned in the production process, therefore a high will reduce the productivity of the fertilizer company. The hardness of fertilizer granules is important in relation to caking in that weak granules may deform and fragment, causing increased points of contact with other granules and resulting in an increase in caking propensity and dust formation (Walker *et al.* 1998) Granule fertilizer products must be quite hard so that it does not break easily and creation of excessive dust during handling (IFDC 1998).

Filter cake is industrial waste produced of sugar factories. Mostly, filter cake is composed of clay and contains dispersed of organic colloid (Prasad 1976). Kuswuri (2009) states that among sugar factory wastes, filter cake has the highest pollution grade and creates a problem for sugar factory and its surroundings. The percentage filter cake produced is approximately 4-5% weight of sugar cane stems per hectare.

The objectives of this study are assessing filter cake utilization as a filler in the production of compound fertilizer and assessing the influence of filter cake addition to the granule hardness (crushing strength). This study aims to analyze the filter cake

utilization as material substitution for clay in compound granule fertilizer of 15-15-15 + 5S for particle size distribution and granule hardness. It is expected to obtain scientific data about granular compound fertilizers properties as effect of addition of filter cake as organic compounds mixed with clay as standard filler.

MATERIALS AND METHODS

Materials and Apparatus

The apparatus which was used in this study were pan granulator, water sprayer, digital scale, crusher, oven, sieve shaker, stopwatch, spatula, crushing strength gauge. Some materials which used in this study were Urea, DAP (Diamonium phosphate), KCl, SOP (K_2SO_4), Ammonium sulfate, clay, lignosulfonate, filter cake and water.

Experimental Procedure

The granulation test was conducted based on NPK formula 15-15-15 + 5S with variables of binder materials composition and K_2O material sources. The binder composition variable was based on filter cake composition which divided into 5 there are (filter cake : clay): 60: 40, 70: 30, 80: 20, 90: 10 and 100: 0%. Detail of combination can be seen in table 1. Each treatment variation was carried out three times.

Granulation Process

Wet granulation process was conducted in using pan granulator which has capacity 2 kg / batch, 23 rpm rotation speed and 50° pan incline (Litster and Ennis 2004; Gluba and Obraniak 2011). The first step : raw material was weighed according to the theoretical consumption and then mixed homogeneously. The process then proceed in agglomeration as long as 15 minutes and drying for 25 minutes. Pan granulator was equipped with a manual scrapper to avoid sticking materials in the pan granulator body. During the agglomeration process, water was added as a liquid binder to wet and to assist the agglomeration process (Obraniak and Gluba 2011).

Table 1. Combinations of treatments with source K and percentage filter cake

Variable	Source of K_2O	Filler	
		%Filter cake	% Clay
K1B0	KCl	0	100
K1B1	KCl	60	40
K1B2	KCl	70	30
K1B3	KCl	80	20
K1B4	KCl	90	10
K1B5	KCl	100	0
K2B0	K_2SO_4	0	100
K2B1	K_2SO_4	60	40
K2B2	K_2SO_4	70	30
K2B3	K_2SO_4	80	20
K2B4	K_2SO_4	90	10
K2B5	K_2SO_4	100	0



Figure 1. Compression tester for measuring granule crushing strength

Crushing Strength

Crushing strength is measured for each granule. Measurements were done on granules product at 2-4 mm diameter which is a standard size of granular fertilizer. This test uses 15-20 granules of the same size (Hignett 1985). Measurements were carried out using a compression tester Mark 10 as in Figure 1. First place the fertilizer granules to be tested into the tool pan tester. Then set the scale needle on the compression tester to zero position. Following, presser give to the granul fertilizer, and note the energy needed to destroy every fertilizer granules.

Data processing

Effect of treatment on size distribution and hardness of the granules was performed by an analysis of variance and Least Significant Difference (LSD) at 1 and 5% test level.

RESULTS AND DISCUSSIONS

1. Granule particle size distribution

Granule size distribution analysis was carried out to assess the effectiveness of the fertilizer granulation process. The greater value of on size granule product (size 2-4 mm) indicates that raw fertilizer mixture materials was chategorized tend to easy agglomeration, while if the greater percentage of undersize granule products classified as difficult agglomeration. The results of the analysis using the sieve shaker equipment show the size of 2-4 mm diameter of granule is the most dominant (Table 2), appearance of the results of on size granule products in various treatments can be seen in figure 1. Oversize product represents that the granulating process in several parts of raw materials running well. Increased granule size occurs due to more wetting in the granulation process in the pan granulator . Addition of liquid to the raw materials initiate the growth of initial granule (Obraniak and Gluba 2017).

Product Quality Testing

Percentage of granule of 2-4 mm size

The percentage of granules of 2 - 4 mm size was measured using a sieve shaker, a sieve size of 4, 5, 10, 12 US mesh with an amplitude setting of 2 running for 5 minutes. The percentage of granule size is calculated by the formula (Mangwandi *et al.* 2012):

$$\%UG = \frac{M1}{M2} \times 100\% \quad (1)$$

where:

- % UG = percentage of granule size of 2-4 mm (%)
- M1 = mass of compound fertilizer 2-4 mm size (g)
- M2 = mass of total fertilizer (g)



Figure 1. Compound fertilizer products

Table 2. Size distribution of 15-15-15 + 5S granule products in various treatments

Variable	Size Distribution		
	Onsize	Under size	Over size
	2-4 mm	< 2 mm	> 4 mm
K1B0	90.16 c	7.45 a	2.39 a
K1B1	77.84 bc	19.72 a	2.44 a
K1B2	77.30 bc	18.54 a	4.17 a
K1B3	69.75 ab	20.07 a	10.18 a
K1B4	68.92 ab	15.07 a	16.01 a
K1B5	61.41 ab	18.36 a	20.24 a
K2B0	78.76 bc	10.63 a	10.61 a
K2B1	72.96 abc	9.14 a	17.90 a
K2B2	76.45 bc	20.31 a	3.24 a
K2B3	71.94 ab	20.59 a	7.47 a
K2B4	68.74 ab	29.40 a	1.87 a
K2B5	56.75 a	26.21 a	17.04 a

The results of the granule size distribution test showed that the highest percentage of on sized granules was achieved in the K1B0 treatment (100% clay filler, and KCl as source of K) followed by K2B0, K1B1, K1B2 treatments successively. Statistically the K1B0 treatment was significantly different from the other treatments based on LSD test. Lower percentage of under sized granule were also found in the K1B0 treatment followed by K2B0, K1B1, K1B2 treatments successively, but statistically not significant.

Overall, the granulation process is increasingly difficult as the clay component decreases in the binder. Until now, clay was generally the main binding agent in the wet granulation process of compound fertilizer. The results of research show that, in some cases the salt bridges in the granulation process increase with the addition of insoluble binders such as clay or phosphate rock in the material mixture (IFDC, 1998). Table 2 showed the treatment of K1 (KCl as K₂O source) percentage of the size distribution on size is better.

Table 3. Chemical composition of compound fertilizer granules with source raw material urea, DAP, ZA, KCl, clay and filter-cake

Composition	unit	K1B0	K1B1	K1B2	K1B3	K1B4	K1B5
Total Nitrogen	% wt	15.66	15.52	15.60	14.93	15.32	15.40
N-NH ₄ ⁺	% wt	11.17	10.87	11.28	10.81	10.88	10.70
N-NO ₃ ⁻	% wt	0.60	0.53	0.41	0.13	0.40	0.49
Total P, as P ₂ O ₅	% wt	14.74	15.67	15.82	14.45	14.79	15.27
P ₂ O ₅ Citric acid Sol.	% wt	11.18	11.19	10.36	10.34	10.75	10.01
P ₂ O ₅ water sol	% wt	12.46	13.43	13.03	12.77	12.50	12.24
Potassium, as K ₂ O	% wt	16.69	16.62	15.67	16.32	16.30	16.47
K ₂ O Citric acid Sol.	% wt	16.67	15.31	15.16	15.37	15.03	15.90
K ₂ O water sol	% wt	15.58	15.23	14.75	14.81	15.12	15.09
Sulfur	% wt	4.45	4.27	4.36	4.39	4.11	4.13
Cl	% wt	13.78	15.22	14.48	17.13	16.90	14.55
CaO	% wt	0.98	0.39	1.01	1.22	1.72	1.82
Fe ₂ O ₃	% wt	2.42	2.87	1.96	1.73	1.99	1.80
Al ₂ O ₃	% wt	3.66	4.79	2.83	2.44	2.96	2.22
SiO ₂	% wt	5.46	6.64	4.70	4.24	4.96	4.02
MgO	% wt	1.72	1.43	1.46	1.67	1.99	1.90
Moisture, H ₂ O	% wt	2.03	2.84	3.08	2.30	2.83	2.91

Table 4. Chemical composition of compound fertilizer granules with source raw material urea, DAP, K₂SO₄, clay and filter-cake

Composition	Unit	K2B0	K2B1	K2B2	K2B3	K2B4	K2B5
Total Nitrogen	% wt	15.06	16.08	17.46	16.56	16.33	17.36
N-NH ₄ ⁺	% wt	7.27	7.32	6.79	7.23	7.2	7.39
N-NO ₃ ⁻	% wt	0.73	0.98	1.03	1.2	1.29	0.63
Total P, as P ₂ O ₅	% wt	15.73	15.05	15.06	15.25	15.45	14.64
P ₂ O ₅ Citric acid Sol.	% wt	10.96	10.56	10.31	11.22	10.99	10.31
P ₂ O ₅ water sol	% wt	12.27	12.93	12.35	12.86	12.62	12.61
Potassium, as K ₂ O	% wt	17.22	15.19	16.92	16.23	17.5	16.54
K ₂ O Citric acid Sol.	% wt	15.28	15.36	16.3	15.6	15.76	15.26
K ₂ O water sol	% wt	14.6	14.46	14.92	15.19	15.91	15.71
Sulfur	% wt	4.74	4.62	5.03	4.89	5.25	4.82
Cl	% wt	2.45	2.53	2.23	2.32	2.38	2.53
CaO	% wt	0.80	1.52	3.00	3.57	3.62	4.50
Fe ₂ O ₃	% wt	6.63	2.63	5.08	4.88	3.39	3.18
Al ₂ O ₃	% wt	13.28	5.42	6.69	5.11	5.71	3.79
SiO ₂	% wt	16.31	7.45	9.29	8.75	10.10	9.16
MgO	% wt	2.70	1.80	2.06	2.09	2.04	2.20
Moisture, H ₂ O	% wt	1.65	1.27	2.06	2.4	3.75	2.31

The K1 treatment, to achieve S content in the formulation, it is added Ammonium sulfate (ZA) The addition of ammonium sulfate contributes speed up the granulation process. This is in line with research conducted by Xue *et al.* (2017) which resulted the addition of ammonium sulfate by 1-2%wt significantly increased the granulation speed. The results of the chemical composition analysis of the granule products showed that the ammonium content in the K1 treatment was higher than K2 (See Table 3 and 4). Moreover, granulation in K1 resulted in a gradual oversized granule product in line with filter cake increasing value while in treatment K2 there was no clear trend. However, the undersize product in the treatment K2 is quite high when compared treatment K1.

2. Crushing strength

The mechanical crush strength values of the granules in various treatments are presented in Figure 2. This figure shows the effect of granule fertilizer size (2-4 mm and 1.7-2 mm) on the hardness in various treatment. The results of the study show that the crushing strength of the product increases with diameter of NPK particle size increment. while the values of the hardness of compound products on standard granule sizes are presented in Table 5. Overall, the fertilizer hardness value is acceptable. The hardness of the product in the treatment using KCl as a source of K is better than using K₂SO₄. This is due to the presence of ammonium and nitrate content form urea reacts with KCl to form ammonium chloride (see table 3 and 4). Because a saturated ammonium chloride

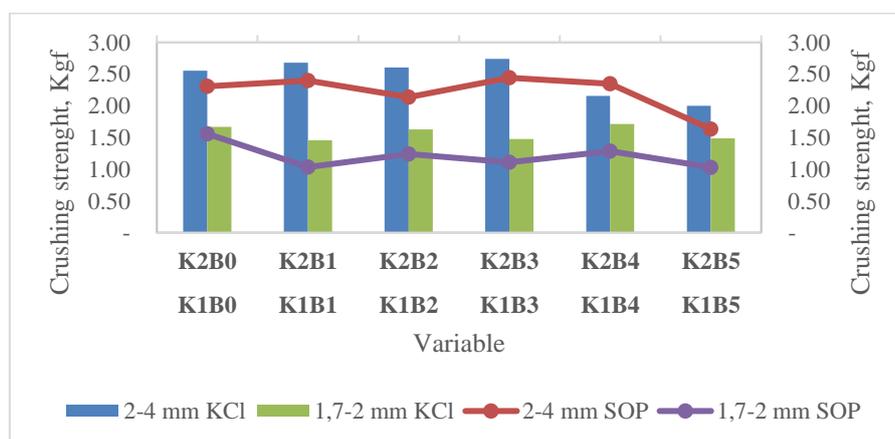


Figure 2. Graph of crushing strength compound fertilizer product

Table 5. Value of Crushing strength compound fertilizer product

Variable	Crushing strength		Remark *)
	2-4 mm Kgf		
K1B0	2.56 a		Acceptable
K1B1	2.68 a		Acceptable
K1B2	2.60 a		Acceptable
K1B3	2.74 a		Acceptable
K1B4	2.15 a		Need concern
K1B5	2.00 a		Need concern
K2B0	2.30 a		Need concern
K2B1	2.40 a		Need concern
K2B2	2.14 a		Need concern
K2B3	2.44 a		Need concern
K2B4	2.35 a		Need concern
K2B5	1.63 a		Need concern

solution on the external surface of granules evaporates to form crystal bridges with adjacent particles, so that the bond between particles of raw material becomes stronger. The highest value of product hardness is obtained in the treatment of K1B3 (KCl as the source of K; Filter cake: clay 80: 20%). However statistically was not significantly different in all treatments.

Higher the value of NPK granule products crushing strength, the shelf life of the product will be longer. Hereafter the product is not easily crushed during storage.

CONCLUSIONS

The results of the research showed that statistically the combination of filter cake and clay 70:30 had a size distribution and hardness of granules not significantly different from standard fertilizer (100% clay). The crushing strength of the product in the variable using KCl as a source of K is better than using K₂SO₄.

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