Moisture Formation in Urea Magnesium Sulphate Synthesized Via Ball Milling Technique

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Abstract— **Higher population urge agricultural family to rise up their production in food source along with maintaining the growth of the plant as well. In order to improve nutrient in the soil so that the plant able to growth well, mechanochemical method was practiced as to synthesis of Urea Magnesium Sulphate (UMS). The UMS was synthesized based on different ratio and milling time via ball milling and the moisture content is investigated on the sample. The synthesizing is conducted based on ratio 1:1, 2:1, 1:0.7 and 1:2 of magnesium sulphate to urea with different time milling of 2, 4 and 8 minutes. The characterization of UMS is analyze by using X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) is to determine the functional group of UMS and Thermal Gravimetric Analysis (TGA) is used to identify the moisture formation in UMS. It was found that most of the sample crystallized based on XRD graph pattern. The analysis found that urea magnesium sulphate with ratio of 1:2 at 8 minutes milling time has synthesized urea magnesium sulphate. While based on the FTIR the range of IR Spectrometry adsorption between 515cm1 to 4000cm-1 and it is shown that the most of the bond is found and similar with each other in the sample. To study proximate analysis, TGA is used as to identify the moisture content which is in between 800C to 1500C. High amount of magnesium sulphate and longest milling time has the lowest amount of moisture content. As to ensure the urea magnesium sulphate is synthesized with low moisture content, the milling time should be conducted with longer period.**

Keywords— Fertilizer, Urea Magnesium Sulphate, Ball Milling, XRD, FTIR, TGA

I. INTRODUCTION

Urea is well known in preferred fertilizer and widely used in agricultural industry by involving in supplying nutrient to soil such as crops [7]. Fertilizer is used as additive in the soil as to improve the levels of nutrient in plant and improve the chemical and physical properties f soils as well [13]. Crystalline urea has the highest amount of nitrogen content in solid fertilizer ad it is the most popular in the developing country. Urea is considered as favorable to the soil because urea itself involve with contribution in supplying nutrient for plant growth which is nitrogen, phosphorus and potassium (N-P-K) [11].

The characteristic of urea which is naturally decomposed to ammonia causes gaseous nitrogen loss will occur and can be up to 50%. The nature of urea in highly hygroscopic at high humidity also one of the reason for the ammonia losses [7]. Hygroscopycity in urea fertilizer provide tendency to the fertilizer to absorb moisture in the air. Due to its hygroscopic properties it is not only influences the physical properties but also affected the performance of the fertilizer during the absorption to the soil such as the flow ability [7]. Inorganic salt such as magnesium sulphate in urea can help urea in reducing ammonia losses to surrounding [10][14].

The presence of magnesium and Sulphur with urea in the fertilizer could help in providing primary and secondary nutrient in the soil [6]. Magnesium sulphate can help plant to improve the deficiency in magnesium for soil because magnesium can improvise chlorophyll production since magnesium sulphate also play roles as fertilizer additive [5]. Sulphur supply vitamins, amino acids and enzyme to assist in growing plants. From this constituent of mixed fertilizer, the plant growth can be improved as well [5].

Urea magnesium sulphate can be prepared from grinding, compaction and mixing reagent in the presence of physical water or any solvent [5][14]. Synthesizing of urea magnesium sulphate also can be prepared by solvent free with mechanical works and the reaction happens between the materials called as mechanochemistry [12].

This reaction is a chemical reaction that induces by the direct absorption of mechanical energy which is comes from the high energy ball milling [8]. Ball milling process is when a mixture of chemical substance powder that is placed in a milling jar and it is subjected to high energy impact from the balls during the process. High energy ball milling is one of the common ways to reduce particle size from grinding in a shorter time and may allow synthesis of a new materials [1].

Ball milling providing the green chemistry effect in the synthesizing material due to an approaching in solvent free during the production of new product. Other than that, this mechanical work potentially in prevention of waste and less hazardous chemical synthesis. 3 significant variable that affect the reaction of the compound can be classified into chemical, technology and process parameters.

This research is conducted to investigate the moisture content in the synthesis of urea magnesium sulphate via ball milling technique.

II. METHODOLOGY

A. Materials

Raw material that being study is granulated urea (manufactured by Petronas) and magnesium sulphate (manufactured by R&M Chemicals). These raw materials are used to synthesis of urea magnesium sulphate.

B. *Milling Process*

The urea granules were crushed into powder by using mortar and the powder with magnesium sulphate are weight based on different ration. 7 stainless steel ball were used and the weight ratio for ball to powder is kept to 10:1. Mass of the urea and magnesium sulphate is weighted based on the ratio of magnesium sulphate to urea which are respectively set up to 2:1, 1:1, 1:0.7, and 1:2. Synthesized is carried out using Retsch laboratory scale mixer ball (Model: MM400). The stainless mill jar is filled with the stainless steel ball and the mixture of urea and magnesium sulphate. The milling process is fixed with 30Hz of frequency while varying the milling time and the powder ratio. Milling time are set at 2, 4 and 8 minutes for each sample

C. *Characterization*

The X-ray diffraction (XRD) (Model: ExpertPRO) is used for phase identification of a crystalline material and provide detailed information about unit cell dimensions. The sample is operated over a 2 theta range of 10^o to 90^o. The samples also analyzed by using Fourier Transform Infrared Spectroscopy (FTIR) (Model: Spectrum One) which is to identify the functional group in the urea magnesium sulphate. The FTIR spectrum is running in the range 400-4000 cm^{-1}. FTIR will guide in determining the molecular structure of urea magnesium sulphate. Thermal Gravimetric Analysis (TGA) (Model: TGA/DSC 1 Star System) is used to measure the weight of sample after it is decomposed when heated at constant rate. The analysis is carried out from 30^oC to 1200^oC at a heating rate of 10^oC min^{-1} with the flowing of nitrogen gas at 20ml min^{-1}.

III. RESULTS AND DISCUSSION

A. *The physical observation*

The appearance for the urea magnesium sulphate based on different ratio displays different physical condition. High ratio of magnesium sulphate give a hard powdery texture compared to high amount of urea in the milling process. For ratio of 1:2, the physical properties can be seen that the sample have more moisture content. The milling time does not give any effect on the appearance for the same ratio but different ratio does affect the physical condition of the urea magnesium sulphate. Figure 1 shows the physical condition for the ratio of 2:1 and 1:2 of magnesium sulphate to urea at 8 minutes. It is shown that high amount of urea content during milling process giving a wet and sticky due to highly hygroscopic properties which have tendency to absorb the water in the air. Hygroscopicity also can caused the sample to be clumpy and has low fluidity [12].

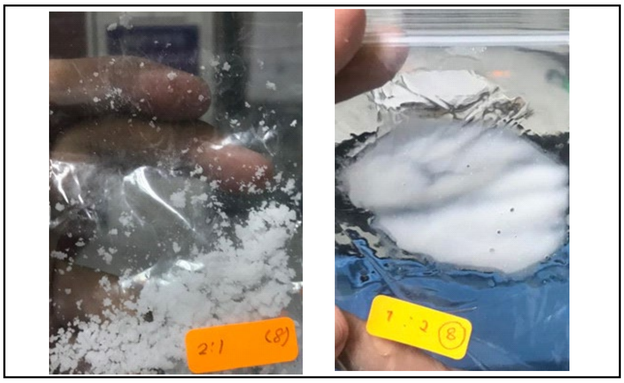

Figure 1: The physical condition of Urea Magnesium Sulphate between ratio of 2:1 and 1:2 at 8 minutes

For the ratio of 1:1 and 1:0.7 at the same milling time, 8 minutes, the physical properties of the sample is tend to be sticky for ratio 1:1 due to high amount of urea content compared to 1:0.7 while the appearance for 1:0.7 is in between crystalline but still sticky because the amount of magnesium sulphate not sufficient enough to balance between moist texture and hard texture. Therefore this magnesium sulphate cannot reduce the stickiness of the urea magnesium sulphate.

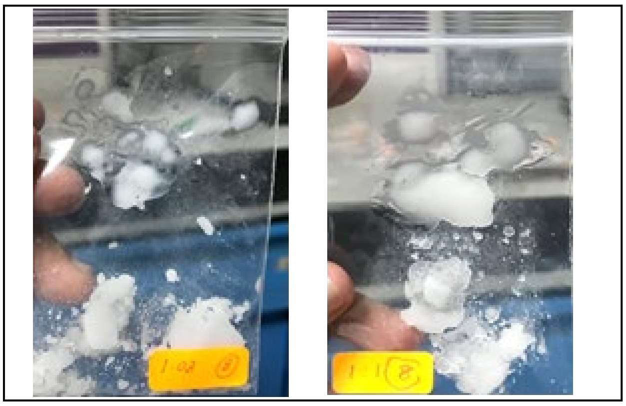

Figure 2: The physical condition of Urea Magnesium Sulphate between ratio of 1:0.7 and 1:1 at 8 minutes

B. *X-ray Diffraction (VRD)*

The X-ray diffraction peak of the urea magnesium samples are respectively at figure 3, figure.4 and figure 5 for 2 minutes, 4 minutes and 8 minutes of milling time

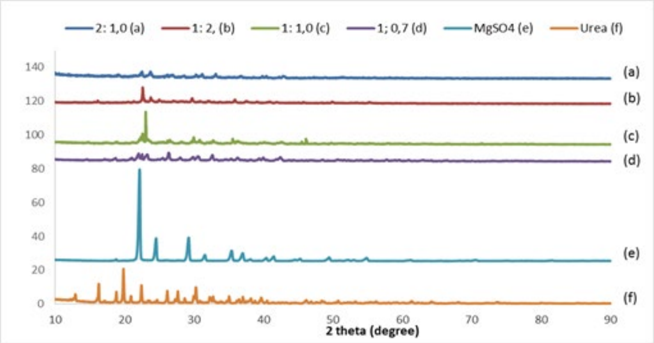

Figure 3: The X-ray Diffraction (XRD) analysis of Urea, Magnesium Sulphate and Urea Magnesium Sulphate at different ratio for 2 minutes

Based on Figure 3, all of the ratio for the compound between urea and magnesium sulphate have a crystalline characteristics due to the pattern of the peak in between 20^0 – 30^0. The sharp peaks on the pattern determine the characteristics of the compound which is crystalline compound. Peak pattern in between 20^0 – 30^0 for the compound appears to have similarities with the raw material. It can be clearly seen especially for ratio 1:1 and 1:2. Both ratio have the same sharp peak pattern with urea at 22.421^0. This is due to the urea content is slightly higher compare to the content of urea magnesium sulphate. Therefore, the both peak can be analyzed as urea's peak. While for the analysis on the ratio of 2:1 and 1:0.7, the peak pattern for the both ratio is appear at 29.405^0. Ratio of 1:0.7 and 2:1 have the slightly identical peak pattern with raw magnesium sulphate. Unfortunately the magnesium sulphate's peak at the both ratio is not as sharp as the raw material therefore the location of the peak also can be assumed that it is match with urea's peak. Due to the urea's content in the both ratio is slightly lower than magnesium sulphate content, it can be assumed that the peak at both ratio can be justified that it is for magnesium sulphate. Since the peak is no too obvious as the raw materials' peaks, the presence of the raw could be dissolve in the reaction during milling time in the ball mill. Thus, for 2 minutes of milling time, the milling time is not sufficient enough for the reaction to occur.

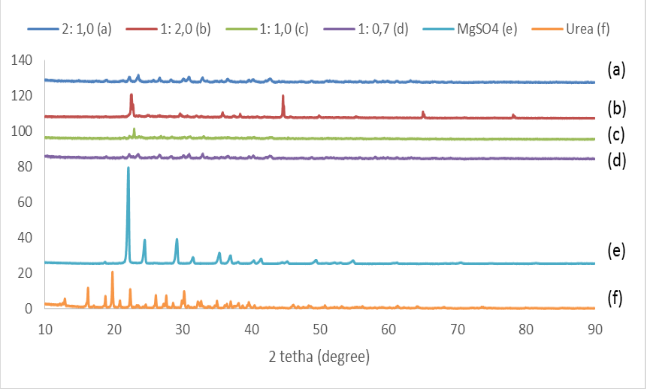

Figure 4: X-ray Diffraction (XRD) analysis of Urea, Magnesium Sulphate and Urea Magnesium Sulphate at different ratio for 4 minutes

Based on figure 4, the graph pattern for all ratios similar with the graph pattern at 2 minutes. For the ratio of 1:0.7 and 2:1, they have similar location of the peak and the pattern with the same ratio at 2 minutes. Even longer time is conducted, there is still no changes for the both ratio. So it still can assume that the peaks which is belongs to the magnesium sulphate due to the higher content in magnesium sulphate is dissolved because the reaction is occur in the ball mill. The peak of urea or magnesium sulphate especially cannot be detected at any range of peak pattern. Regarding the urea magnesium sulphate at the ratio of 1:2 the presence of peak in between 20^0 - 30^0 is slightly the same with the previous peak pattern at 2 minutes with the presence of urea content where it is located at 22.421^0 but not available at the ratio of 1:1. The nearest peak which located with urea's peak is situated at 22.843^0 for the ratio of 1:1. Based on the observation, this peak consider to be the presence of urea because urea tend to have more sharp peak compared with magnesium sulphate at this location. Synthesizing of urea magnesium sulphate does not resemble at all ratio for 4 minutes but urea magnesium sulphate at the ratio of 1:2 does not have similar peak compare to the other ratios. Urea magnesium sulphate at ratio 1:2 have a new peak at 44.640^0 which is not identical with any raw material. This sharp peak is a new finding in this ratio and is can be justified as a new product since mechanochemistry is applied during milling time. The reaction occur at this ratio and time therefore, this peak is the version of urea magnesium sulphate.

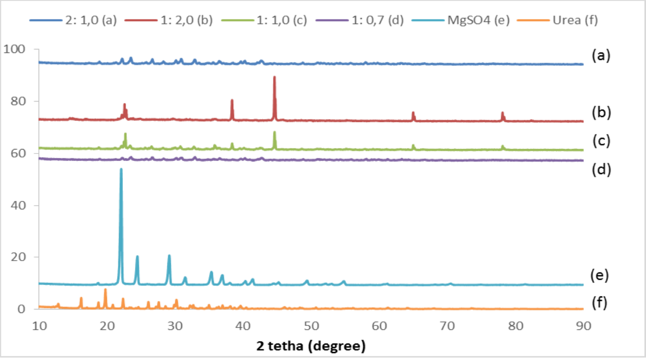

Figure 5: X-ray Diffraction (XRD) analysis of Urea, Magnesium Sulphate and Urea Magnesium Sulphate at different ratio for 8 minutes

Based on figure 5, the peak pattern for all ratios still can be classified as crystalline structure due to the sharp peak pattern at all ratios. Urea magnesium sulphate at ratio 1:1 and 1:2 drift to have alike peak pattern compare to the ratio of 1:0.7 and 2:1. The presence of urea still available at the ratio of 1:1 and 1:2 at 22.812^0 and 22.693^0 respectively but in between 35^0 – 50^0 at both ratio, the pattern is appear have the new sharp peak where it is located at 44.657^0 for the both ratios. The location for the new peak is the same which means that both ratios capable to under mechanosynthesis which is synthesis of urea magnesium sulphate is developed. 8 minutes for the both ratios is possible to experiences mechanochemistry in ball milling because the new peaks only appear when the energy is sufficient enough for the balls to collides each other and improvise the kinetic energy then promote a synthesis in the ball mill. For the ratio of 1:0.7 and 2:1 at 8 minutes, both still have the same pattern as 4 minutes and 2 minutes where the raw materials pattern still cannot be seen at the graph pattern which the reaction occur because the peaks from the raw material is no longer in existence but the synthesizing of urea magnesium sulphate does not occur. The possibility of these case is due to the insufficient milling time or the ratio is not is not the right amount for synthesizing of urea magnesium sulphate.

Basically, the structure characteristics for all the ratios is the same. All of the sample classified as a crystalline compound because the presence of sharp peaks in every graph. Even though the physical appearance for certain ratio give a moist, wet or clumpy, the product still characterized as crystalline compound. But after synthesizing between urea and magnesium sulphate, the presence for the both raw materials cannot be detected together because the reaction is still occur for urea and magnesium sulphate to react together. High ratio in urea only can detect urea but cannot detect magnesium sulphate and vise versa. The possibility of this condition might be cause from the insufficient milling time. Short milling time will make the mechanochemistry not going to happen. The reaction between urea and magnesium sulphate is not occur at all ratios because the residence time for urea magnesium sulphate might be longer than 8 minutes. The mechanosynthesis only occur after 4 minutes at the ratio of 1:1 and 1:2 which is higher amount of urea content. Due to the nature of urea which is highly hygroscopic, the tendency for synthesizing of urea magnesium sulphate to occur is high.

The reagent ratio play certain parts in synthesizing urea magnesium sulphate and to increase the efficiency of milling process is mostly depending on the technology parameter such as size, number of milling balls. Process parameter such as operating frequency contribute in energy controlling. When the frequency of mill increase, the kinetic energy between the ball and the veil also will increase causing the chemical reaction to develop. During the process of high energy from the milling causing the high energetic impact between the milling pot and the ball to the powder. High energy ball mill can imposed to the balls then could correspond to a kinetic energy and could rise the temperature of a certain particle inside the mill and it is causing from the interdiffusion between layers of crystalline defect which induced by the milling action. From the increasing in kinetic energy and bulk temperature that composed from the high energy can promote a synthesis of the compound inside the veil by increased the surfaced energy and defectivity. Large energy that computes by the mechanical effect from the balls and the veils makes high energy ball mills able to undergo the synthesis by reaction and form a new product which is urea magnesium sulphate [3][4][9].

C. *Fourier Transform Infrared Spectroscopy (FTIR)*

The FTIR spectrum of the urea, magnesium and urea magnesium sulphate was recorded in range of 515 cm-1 to 4000 cm-1 for urea magnesium sulphate with the ratio of 1:0.7, 1:1, 1:2 and 2:1 for 2, 4 and 8 minutes. The wavenumber for the peak at every ratio is recorded in table 2, 3 and 4 for 2 minutes, 4 minutes and 8 minutes respectively

Table 1: FTIR analysis of urea magnesium sulphate

Raw Material	Urea	Magnesium Sulphate	Funtional Group
IR Spectrum Wavenumber (cm^{-1})	3750	3750	(O-H)with Stretching Mode
		3394	H_2O molecule (freewater symmetric stretch)
	3428	-	amines (N-H) with strong-medium peak(symetric stretch, strretching vibration)
	3327	-	amines (N-H) with strong-medium peak(symetric stretch, strretching vibration)
	-	1743	carbonyl C=O with asymmetric stretching vibration
	-	1630	aromatic C=C with asymmetric stretching vibration
	1587	1541	aromatic C=C with asymmetric stretching vibration
	1457	1456	N-C-N (asymmetric stretch, stretching vibration)
		1084	SO_4^{2-} (asymetric stretch, stretching vibration)
	787		C-H with aromatic stretching vibration
		599	SO ^{2} (bending mode)

Table 1 shows the summarization of FTIR spectra for urea and magnesium sulphate. These absorbance value represent the presence of functional group in each samples and as the indicator for the milling compound to be analyzed.

Table 2: FTIR analysis of Urea Magnesium Sulphate at different ratios for 2 minutes milling time

Urea Magnesium Sulphate (ratio)	1:0.7	1:1	1:2	2:1	Functional Group
IR Spectrum Wavenumber (cm $^{-1}$)	3324	3341	3337	-	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	3193	3196	3201	3210	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	2169	2161	2160	2210	alkyne (C=C) with medium - varaible peak (stretching vibration)
	1595	1595	1590	1595	amide II (N-H) deformation (bending mode)
	1474	1464	1461	1473	N-C-N (asymmetric stretch, stretching vibration)
	1067	1078	1076	1071	SO ^{2} (asymetric stretch, stretching vibration)
	-	-	779	-	Mg-O (stretching vibration)
	-	-	583	-	SO ^{2} (bending mode)

Table 2 represent for 2 minutes of milling time by recording the following ratio of 1:0.7, 1:1, 1:2 and 2:1. FTIR had analyzed the presence of amines group (N-H) with broad envelope at 3324cm^{-1} and 3193cm^{-1} for ratio of 1:0.7, 3341 cm^{-1} and 3196 cm^{-1} for ratio of 1:1, 3337 cm^{-1} and 3201 cm^{-1} for ratio of 1:2 and 3210 cm^{-1} for ratio of 2:1 urea magnesium sulphate with stretching vibration. The variable intensity peak with stretching vibration of C=C is observed at all ratios where which positioned at 2169 cm^{-1}, 2161 cm^{-1}, 2160 cm^{-1}, and 2210 cm^{-1}. There are deformation by bending mode of secondary amide are observed at 1594 cm^{-1}, 1590 cm^{-1}, 1591 cm^{-1} and 1597 cm^{-1} with the ratio of 1:0.7, 1:1, 1:2 and 2:1 respectively. Sharp peak of N-H-N with stretching vibration has been observed at 1474 cm^{-1}, 1464 cm^{-1}, 1461 cm^{-1} and 1473 cm^{-1} for respective ratio. The band at 1067 cm^{-1}, 1078 cm^{-1}, 1076 cm^{-1} and 1071 cm^{-1} is cause by the SO_4^{2-} asymmetric stretching vibrations. The small intensity of peak pattern at 779 cm^{-1} only appears at the ratio of 1:2 and it is observed as the Mg-O stretching mode and the same ratio observed the band at 583 cm^{-1} which is due to the presence of SO_4^{2-}.

Table 3: FTIR analysis of Urea Magnesium Sulphate at different ratios for 4 minutes milling time

Urea Magnesium Sulphate (ratio)	1:0.7	1:1	1:2	2:1	Functional Group
IR Spectrum Wavenumber (cm $^{-1}$)	3334	3337	3334	3339	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	3194	3202	3199	3195	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	2186	2229	2171	2154	alkyne (C=C) with medium - varaible peak (stretching vibration)
	1594	1590	1591	1597	amide II (N-H) deformation (bending mode)
	1468	1464	1457	1478	N-C-N (asymmetric stretch, stretching vibration)
	1068	1077	1070	1075	SO ^{2} (asymetric stretch, stretching vibration)
	-	-	-	786	Mg-O (stretching vibration)
	-	586	-	590	SO ^{2} (bending mode)

Table 3 represent the analyzed FTIR for 4 minutes milling time. The frequency of 3334cm^{-1} and 3194 for ratio 1:0.7, 3337cm^{-1} and 3202 cm^{-1} for ratio of 1:1 3334 cm^{-1} and 3199 cm^{-1} for ratio 1:2 and, 3339 cm^{-1} with 3195 cm^{-1} is due to symmetric N-H vibration of urea magnesium sulphate. The alkyne group (C=C) with variable intensity peak is detected at all ratios of 1:0.7, 1:1, 1:2 and 2:1 where is it located at 2186 cm^{-1}, 2229 cm^{-1}, 2171 cm^{-1}, and 2154 cm^{-1} respectively. The strong peak that known as amide secondary band respectively that mean N-H deformation at peak pattern of 1594 cm^{-1}, 1590 cm^{-1}, 1591 cm^{-1} and 1597 cm^{-1} for ratio of 1:0.7, 1:1, 1:2 and 2:1 respectively. The strong intense of peak pattern at 1068 cm^{-1} of 1:0.7, 1077 cm^{-1} of urea magnesium sulphate at ratio of 1:1,1070 cm^{-1} of urea magnesium sulphate with ratio of 1:2 and 1075 cm^{-1} for ratio of 2:1 is observed to SO_4^{2-} asymmetric stretching vibrations. Mg-O with stretching mode only appears at ratio of 2:1 with intensity of 786cm^{-1} and bending mode of SO_4^{2-} is observe at 586cm^{-1} and 590cm^{-1} at ratio of 1:1 and 2:1.

Table 1: FTIR analysis of Urea Magnesium Sulphate at different ratios for 8 minutes milling time

Urea Magnesium Sulphate (ratio)	1:0.7	1:1	1:2	2:1	Functional Group
IR Spectrum Wavenumber (cm $^{-1}$)	-	3328	3340	3339	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	3194	3197	3201	3193	amines (N-H) with strong-medium peak (symetric stretch, stretching vibration)
	2189	2159	2159	2199	alkyne (C=C) with medium - varaible peak (stretching vibration)
	1595	1594	1591	1595	amide II (N-H) deformation (bending mode)
	1473	1469	1457	1474	N-C-N (asymmetric stretch, stretching vibration)
	1064	1071	1068	1067	SO ^{2} (asymetric stretch, stretching vibration)
	750	-	-	751	Mg-O (stretching vibration)
	-	-	583	574	SO ^{2} (bending mode)

Table 4 represent the analyzed FTIR for 8 minutes milling time with ratio of 1:0.7, 1:1, 1:2 and 2:1 of urea magnesium sulphate. The broaden peak which is known as symmetric N-H stretching vibration is observed at 3194 cm^{-1} for urea magnesium sulphate by the ratio of 1:07, followed by 3328 cm^{-1} and 3197 cm^{-1} for ratio of 1:1 in urea magnesium sulphate, 3340 cm^{-1} and 3210 cm^{-1} at ratio of 1:2 in urea magnesium sulphate and 3339 cm^{-1} and 3193 cm^{-1} at ratio of 2:1. The alkyne group (C=C) with variable intensity peak is detected at all ratios of 1:0.7, 1:1, 1:2 and 2:1 where is it located at 2189 cm^{-1}, 2159 cm^{-1}, 2159 cm^{-1}, and 2199 cm^{-1} respectively. The strong peak that known as amide II band respectively that mean N-N-H deformation at peak pattern of 1595 cm^{-1}, 1594 cm^{-1}, 1591 cm-1 and 1595 cm-1 at ratio of 1:0.7, 1:1, 1:2 and 2:1 respectively. Asymmetric N-C-N stretching vibration is observe at peak pattern of 1473 cm^{-1}, 1469 cm^{-1}, 1457 cm^{-1} and 1474 cm^{-1}. The strong intense of peak pattern at 1064 cm^{-1} is observed at ratio of 1:0.7 of urea magnesium sulphate, 1071 cm^{-1} of urea magnesium sulphate with ratio of 1:1, 1068 cm^{-1} at urea magnesium sulphate with ratio of 1:2 and 1067 cm^{-1} at ratio of 2:1 is observed to be SO_4^{2-} asymmetric stretching vibrations. The intensity of peak pattern only can be observed at 750 cm^{-1} for urea magnesium sulphate with ratio of 1:0.7 and 751 cm^{-1} for urea magnesium sulphate with ratio of 2:1 by observed the Mg-O for stretching mode. Only 2 ratio that is regarding to the SO_4^{2-} with is bending mode which are 1:2 at 583 cm^{-1} and 2:1 at 574 cm^{-1}.

Based on the analyzed results, it showed that the peak pattern of the compound is shifted from the raw materials' peak. However the observed spectrum is in correspondence with the peaks of urea and magnesium sulphate. The frequency of amine (N-H) and SO_4^{2-} stretching vibration value is decreased compared to the urea's data. This caused by the change of the bond length for the synthesizing by the formation of new bond. The bending vibration of SO_4^{2-} is also been reduced due to the occurrence in bond angle alteration

D. *Thermal Gravimetric Analysis (TGA)*

Thermal gravimetric analysis (TGA) is performed to analyzed moisture content raw materials and the formulated product which is urea magnesium sulphate.

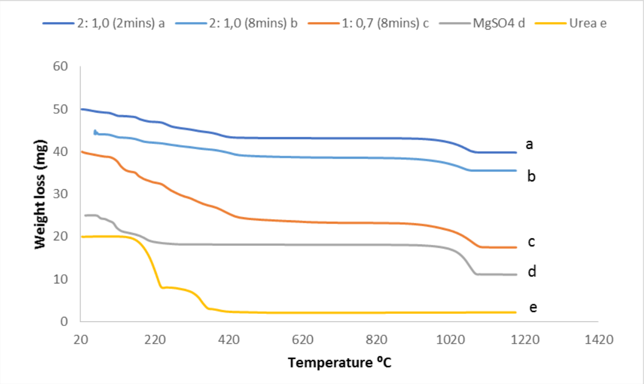

Figure 6: Thermal Gravimetric Analysis (TGA) of Urea, Magnesium Sulphate, 1:0.7 (8 minutes), 2:1 (8 minutes) and 2:1 (2 minutes)

For ratio of 1:0.7 is observed that the urea magnesium sulphate at this milling time and ratio tend to have high degradation compared to the ratio of 2:1 which is it has high amount of magnesium sulphate compare to the other ratio. Magnesium sulphate starts to degrade at temperature of 70^{0}C therefore since the temperature is below the boiling point of water, this sample contain moisture compared to urea, the degradation is starting after 100^{0}C therefore no weight loss at the boiling temperature of water and it can be classified as the moisture of organic matter.

Based on the ratio of 2:1 at 2 different milling time which is at 2 and 8 minutes, both started to degrade before 100^{0}C which is at 70.20^{0}C and 84.43^{0}C for 2 minutes and 8 minutes respectively. Therefore, the moisture content starts to be reduced as the temperature rise up. For ratio of 2:1 at 2 minutes the weight loss is calculated to be 1.3665mg which is about 7.4% of the sample lost the weight compare to the same ratio at different milling time, 8 minutes, the weight loss is 1.13mg approximately about 6% from the initial weight. Same ratio at different milling time could affect the moisture content so based on the sample longer milling time will give low amount of moisture content in the sample where only 6% of the weight sample of 8 minutes milling time is loss which is low amount of moisture is loss after degradation while at 2 minutes milling time loss about 7.4%.

Based on the different ratio at the same milling time also give different amount of moisture content. Basically for ratio of 1:0.7 at 8 minutes, the sample loss about 2.6025mg approximately about 17% compare to the ratio of 2:1 at the same milling time, the weight loss is much lower than ratio of 1:0.7 which is only 7.4%. Urea magnesium sulphate at ratio of 1:0.7 have higher amount of moisture content compare the ratio of 2:1. This situation occur due to the amount of urea is slightly higher at the ratio of 1:0.7 therefore the hygroscopycity in the urea will reduced the weight of the sample. So higher amount of urea will degrade more compare to the low amount of urea.

For the all ratios, the early stage of degradation is starting below the boiling temperature of water which is 100^{0}C. Moisture content starts to be detected below the temperature of 150^{0}C. The weight loss can be detected when the samples start to degrade which is below 100^{0}C. They are still in the range as to detect the moisture content in urea magnesium sulphate. They show the presence of absorbed water molecules in the sample. So the moisture content in magnesium sulphate or urea does affect the urea magnesium sulphate but it still can be consider as the moist from organic compound because naturally urea is the hygroscopic compound so basically urea should have detect the moisture content below the temperature of 100^{0}C.

In between temperature of 150^{0}C-950^{0}C it representing the volatility of the compound and ratio of 1:0.7 is observed to have high volatility compare the ratio of 2:1 because the weight loss for this ratio is bigger than the ratio of 2:1 before it starts to become constant. In other words, the compound at ratio 1:0.7 degrade higher as the physical appearance of this compound is more wet and sticky than the compound at ratio 2:1. Wet compound tend to volatile faster than hard texture compound.

IV. CONCLUSION

In synthesizing of urea magnesium sulphate via ball mill gave different appearance of compound based on different ratio and higher amount urea will give clumpy and sticky form while high amount of magnesium sulphate will give a hard texture to the urea magnesium sulphate. This compound is characterized using x-ray diffraction as to detect the same compound as raw material presence in the urea magnesium sulphate. The presence of new peaks is denote as synthesizing is occur and the presence of same peak as the raw materials peak is denote as the compound is unable to do the complete synthesizing. The best results in milling process according to the analysis, ratio of 1:2 with 8 minutes of milling time able to develop urea magnesium sulphate then followed with ratio of 1:1 with the same milling time. In identifying the functional group FTIR is carried out as to analyze the structural of bond change in the compound. The moisture content is analyzed using thermal gravimetric analysis (TGA) and it shows that there are moisture from the urea magnesium sulphate due to the weight loss at the temperature in between 80^{0}C to 150^{0}C. Low moisture content can be obtained when the amount of magnesium sulphate for milling process I higher than the amount of urea.

Thus, from the ratio 2:1 at 8 minutes milling time has lowest moisture content but according to the XRD analysis, the synthesizing of urea magnesium sulphate does not occur. Therefore as to ensure the synthesizing of urea magnesium sulphate is complete with low moisture content, the milling time is should be conducted longer than 8 minutes. The wet stickiness from urea magnesium sulphate can be supported by analysis from TGA and the synthesizing of urea magnesium sulphate is can be characterized and supported by XRD and FTIR.

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