A Study on the Biogas Production and Waste Recycling from Anaerobic Digestion of Farm's Wastes

Su Su Aung*

Abstract

The production of biogas from farm's wastes such as vegetable wastes and cow dung were studied under anaerobic treatment. The vegetable wastes were collected from Namphar Quarter, Banmaw Township And then, the cow dung was also collected from Kokkodaw Quarter, Banmaw Township, Kachin State. The production of biogas was carried out due to five different conditions using vegetable wastes and cow dung by means of anaerobic digestion within two months. Consequently, each residue after being produced biogas was converted into natural fertilizers. The fertilization activities of residue samples were investigated by standard methods.

Key words : biogas, farm's wastes, fertilization activities, anaerobic treatment

Introduction

Farm's waste

Vegetable wastes generated pose an environmental threat and call for the development of a pollution-free model. Generation of renewable energy by bioconversion of vegetables waste is gaining importance as it has proved to be a proficient means of utilizing the perishable vegetable residues. Researchers and industries are now fully engaged in a number of projects involving the technology of "waste to fuel" with a view to overcome the disposal problems. The present article deals with the studies conducted on vegetables waste for production of various types of biofuel (Johns, 1998).

Uses of cow dung

Cow dung, which is usually a dark brown colour is often used as manure (agricultural fertilizer). If not recycled into the soil by species such as earthworms and dung beetles, cow dung can dry out and remain on the pasture, creating an area of grazing land which is unpalatable to livestock. Cow dung can use to remove oil and grease properties of any waste liquids. In many parts of the developing world and in the past in mountain regions of Europe and in present in backward villages of India, caked and dried cow dung is used as fuel. Dung may also be collected and used to produce biogas to generate electricity and heat. The gas is rich in methane and is used in rural areas of India and Pakistan and elsewhere to provide a renewable and stable source of electricity (Nayono, *et al.*, 2009).

The anaerobic digestion

Anaerobic digestion is controlled biological degradation process which allows efficient capturing & utilization of biogas (approx. 60% methane and 40% carbon dioxide) for energy generation. Anaerobic digestion of farm's waste is achievable but

^{*} Dr, Associate Professor, Department of Chemistry, Banmaw University

different types, composition of farm's waste results in varying degrees of methane yields, and thus the effects of mixing various types of farm's waste and their proportions should be determined on case by case basis. Anaerobic digestion (AD) is a promising method to treat the farm's wastes. While Anaerobic digestion for treatment of animal dung is common in rural parts of developing countries, information on technical and operational feasibilities of the treatment of organic solid waste is limited in those parts. There are many factors affecting the design and performance of anaerobic digestion. Some are related to feedstock characteristics, design of reactors and operation conditions in real time. Physical and chemical characteristics of the organic wastes are important for designing and operating digesters, because they affect the biogas production and process stability during anaerobic digestion. They include moisture content, volatile solids, and nutrient contents. The biodegradability of a feed is indicated by biogas production or methane yield and percentage of solids (total solids or total volatile solids) that are destroyed in the anaerobic digestion. The biogas or methane yield is measured by the amount of biogas or methane that can be produced per unit of volatile solids contained in the feedstock after subjecting it to anaerobic digestion for a sufficient amount of time under a given temperature which is taken to be laboratory temperature (Hilkiah, 2008).

Aim and objectives

Aim

To prepare the biogas and natural fertilizer from vegetable waste and cow dung under anaerobic treatment.

Objectives

- To be chosen farm's waste materials (vegetable wastes and cow dung)
- To produce biogas under anaerobic treatment
- To determine the some physicochemical properties of samples after producing of biogas (residue)
- To determine the total nutrient values of different residues
- To investigate relative composition of prepared natural fertilizers

Materials and Methods

General

All chemicals and reagents used in this research were obtained from Banmaw University. In all the investigations, the recommended and standard procedures of both conventional and modern techniques were employed. Instruments employed in this work consist of lab wares, glass wares and other supporting facilities .The experiments were carried out in the Laboratory of the Department of Chemistry, Banmaw University and the spectroscopic analyses were studied in the University Research Centre, University of Yangon, Department of agriculture (land use) in Yangon and the University Research Centre, Monywa University.

Sample collection

All vegetables waste was collected from Namphar Quarter, Banmaw Township, Kachin State and cow dung was collected from Kokkodaw Quarter, Banmaw Township, Kachin State. All vegetables waste was crushed with knife and slurry was prepared mixing with water.

Sample preparation

All farm's wastes (vegetable wastes and cow dung) were used as semisolid medium. The semisolid medium were prepared as shown following ratios.

 G_1 = Vegetable waste alone (100%)

 G_2 = Vegetable waste (50%) + Cow dung (50%) (1:1) ratio

 G_3 = Vegetable waste (33.3%) + Cow dung (66.6%) (1:2) ratio

G₄= Vegetable waste (66.6%) + Cow dung (33.3%) (2:1) ratio

 G_5 = Only Cow dung (100%)

Anaerobic treatment of farm's wastes for biogas production

In this study, a series of production of biogas and preparation of natural fertilizers were carried out using vegetable wastes and cow dung. Five different conditions were created. The same weight of the sample (50kg) was used in all conditions (Börjesson and Mattiasson, 2007).). Biogas production processes for all selected conditions were carried out simultaneously (Johns, 1998). The particular organic waste was filled into an anaerobic digester. The digester was fixed with (150kPa) rubber tube in the motorcycle pieces was shown in Figure 2. All the experiments were kept under open space for the period of 60 days at ambient temperature. During gas generation, each condition was scanned day by day and week by week (FAO, 2008). In addition, the qualification of liberated biogas was determined by syringe method. When biogas generation was completely over, the residue was converted into natural fertilizers for plant growth study (PANIDA,2013). The results of each experiment were shown in Figure 2 and Table 1.

Characterization and composition of biogas

100mL of dilute sodium hydroxide solution was prepared by dissolving granules of sodium hydroxide in about 100mL of water. 20-30 mL of produced biogas during experiment was taken into the syringe (initially filled syringe with H₂O to reduce air contamination) and put end of the tube into NaOH solution, then pushed out excess gas to gas a 10mL of gas sample. Approximately 20mL of solution was taken and kept the end of the tube submerged in the NaOH solution while shaking syringe for 30 seconds. It was pointed downwards and pushed the excess liquid out, so that syringe level reached 10mL.The volume of liquid (NaOH) solution was read. The results of each experiment were shown in Table 2.



Figure 1. Farm's wastes sampling sites



Figure 2. Production of biogas from farm's wastes

Preparation and physicochemical properties of natural fertilizers from residue samples

The natural fertilizers were prepared by anaerobic treatment of the residue after being produced biogas. The residue from each condition was packed in plastic bag and was buried in the ground for ten weeks. After completely digestion of natural fertilizers, they were allowed to dry in air. Some physicochemical properties of natural fertilizer were investigated by standard methods. And also the relative composition of elements of natural fertilizer from residue samples were determined by ED XRF analysis.

Results and Discussion

Implementation of the research activities, cumulative gas production for different mix ratio (in cm^3) and analytical assay on physicochemical properties of different residues were shown in Table 1 and 2. And then, the nutritive values of different residues were shown in Table 3. Moreover, the relative composition of elements in prepared natural fertilizers were shown in Table 4.

	Produced gas (cm ³) (1-8 weeks)					Average			
Samples	1 st	2 nd	3 rd	4 th	5 th	6 th	7 th	8 th	volume(cm ²)
G-1	1200	4200	3000	1800	1000	400	350	60	1501.25
G-2	800	1250	3000	1000	450	250	150	35	866.875
G-3	1800	3000	4200	1200	600	300	150	10	1406.25
G-4	1800	3600	7200	2400	1200	600	300	150	2156.25
G-5	600	1200	3000	4200	3000	2000	1200	600	1975.00

Table 1.Cumulative Gas Production of Samples During 8 Weeks

In biogas production,G-4 is better than other four tests, it was observed in this experiments the arrangement of biogas production for G-1 to G-5 were 1501.25, 866.875,1406.25, 2156.25 and 1975 cm^3/Vol of digester with comparable gas composition respectively.

 Table 2.
 Physicochemical Properties of Residue Samples (Natural Fertilizers)

Sample	pН	VFA (%)	TS (%)	OM (%)
S-1	6.85	51.78	48.22	0.85
S-2	6.37	61.79	38.21	0.25
S-3	6.31	53.28	46.72	0.36
S-4	6.92	20.10	79.90	0.25
S-5	6.51	40.37	59.63	0.24

(S=Residue sample, TS=Total Solid, VFA= Volatile Fatty Acid, OM =Organic Matter)

S-1= Residue from vegetable waste alone(100%)

S-2= Residue from vegetable waste (50%)+ cow dung(50%) (1:1) ratio

S-3= Residue from vegetable waste (33.3%)+ cow dung(66.6%) (1:2) ratio

S-4= Residue from vegetable waste (66.6%)+ cow dung(33.3%) (2:1) ratio

S-5= Residue from Only cow dung (100%)

The pH of the feed mixture were ranging from 6.31 to 6.92. Volatile fatty acid were ranging from 20.10 to 61.79%. Total solid were from 38.21% to 79.90% and organic matter were 0.85%, 0.25%, 0.36%, 0.25% and 0.24% for S-1 to S-5. The pH values of prepared natural fertilizers are nearly neutral and suitable for growing plants. It

can be known that the greater the total solid, the lesser volatile fatty acid and the more generation of biogas. Organic matter is especially important in providing nitrogen, phosphorous and iron. Moreover, it was observed that the gas began to liberate after running for about 7 days. The gas liberation process was completely over for totally 56 days (8 weeks). The amount of liberated biogas was based on the total weight of 50 kg. The biogas process involves three main steps; hydrolysis, fermentation and methane formation. In the first step, microorganisms, acetogenic bacteria convert organic matter to organic acids possibly decreasing the pH, reducing the methane production rate unless the acids were quickly consumed by the methanogens. pH in the range of 6.31 to 6.92 should be maintained in the anaerobic digestion process which is digestion process is

optimum range for methanogens growths. A number of intermediate products are formed in the next step (fermentation), including alcohols, fatty acids and hydrogen gas. Methane is formed in the last step by a unique group of microorganisms. The raw biogas that is produced consists of mainly methane and carbon dioxide. In this research, the condition G-4 gives the highest yield of biogas.

Sample	Total Nitrogen (%)	Total Phosphorous (%)	Total Potassium (%)
S-1	1.54	11.20	1.89
S-2	1.75	11.29	1.08
S-3	1.19	22.20	1.74
S-4	2.73	22.60	2.14
S-5	2.17	21.40	1.32

 Table 3.
 Analytical Assay of Nutrients in Residue Samples (Natural Fertilizers)

Nitrogen helps plants with rapid growth, increasing seed, fruit production and improving the quality of leaf and forage crops .Total nitrogen percent of S-1, S-2, S-3, S-4 and S-5 were 1.54 %, 1.75%, 1.19%, 2.73% and 2.17 % respectively. Phosphorus that can be absorbed and can made available to meet plant. Plants need phosphorus for strong root growth, fruit, stem and seed development, disease resistance and general plant vigour. Total phosphorus percent of all samples were ranging from 11.20 to 22.60%. Potassium is essential for the life and is present in all soils. The nutrients sometimes called potash, is essential for vigorous growth, disease resistance, fruit and vegetable flavour and development and general plant function. Total potassium percent of all samples were 1.89%, 1.08%, 1.74%, 2.14% and 1.32 % respectively.

No	Element	Natural Fertilizer						
INU	(%)	S-1	S-2	S-3	S-4	S-5		
1	Na	-	0.35	0.34	0.28	-		
2	Mg	-	0.97	1.12	1.15	-		
3	Al	-	13.00	12.00	12.50	8.03		
4	Si	9.34	47.00	37.80	40.20	37.89		
5	Р	-	1.60	2.03	2.13	2.23		
6	S	3.98	1.25	1.91	1.62	1.72		
7	Cl	-	1.06	1.62	1.57	-		
8	K	22.53	22.56	15.65	15.60	21.95		
9	Ca	51.70	51.70	12.50	9.91	6.25		
10	Ti	1.09	1.45	1.61	1.57	1.92		
11	Mn	0.54	0.39	0.53	0.51	1.58		
12	Fe	9.99	9.99	12.20	12.30	17.46		
13	Zn	0.12	0.10	0.12	0.14	0.25		
14	Rb	0.13	0.11	0.16	0.18	0.35		
15	Sr	0.30	0.09	0.14	0.14	0.13		
16	Y	0.01	0.00	0.00	0.00	0.01		
17	Zr	-	0.12	0.12	0.13	-		
18	Rh	-	0.21	-	-	-		
19	Cu	0.04	-	0.04	-	0.08		
20	Ag	0.07	-	-	-	-		
21	Br	0.07	-	-	-	0.06		

Table 4.Relative Composition of Elements in Residue Samples (Natural
Fertilizers) by ED XRF Analysis

The content of calcium in all samples were the highest in S-1 and S-2. Potassium (essential nutrient) content was the second highest in S-1, S-2 and S-5 but silicon was the highest in samples S-2 and S-4.The other elements such as Na,Mg,Al,Fe, Ti, Mn, Br, Al, Cu, Zn, Zr,Rb,Rh,Sr,Y,Cl and Ag were also investigated. All the prepared natural fertilizers from residues do not contain the toxic heavy metals (such as Hg, Pb, Cd and As).



Figure 5. EDXRF spectrum of residue samples

Conclusion

The collected farm's waste (vegetable and cow dung) from Namphar and Kokkodaw Quarter, Banmaw Township, Kachin State were mixed under anaerobic treatment .The biogas was prepared by different mixing ratio of vegetable waste and cow dung G-1 to G-5 at different time intervals. The production of biogas was observed under anaerobic treatment and volumes of the biogas collected were recorded during the experiment period two months. The digestion time is increased, the biogas production is also increased. Among the different mixing ratios of vegetable wastes and cow dung (G-1 to G-5), G-4 was produced more biogas than others four digestion tests. The physicochemical analysis of residue samples, S-1 to S-5 were also investigated .The pH values of all residues possessed near the same.Moreover,S-4 obtained the highest values of total solid(79.90) % and the smallest volatile fatty acid (20.10) % . The total nutrient content and organic matter in all residues were suitable for growing plants. The content of calcium in all samples were the highest in S-1 and S-2. Potassium (essential nutrient) content was the second highest in S-1, S-2 and S-5 but silicon was the highest in samples S-2 and S-4. The other elements such as Na, Mg, Al, Fe, Ti, Mn, Br, Al, Cu, Zn, Zr, Rb, Rh, Sr, Y, Cl and Ag were also investigated by ED XRF. All the prepared organic fertilizers from vegetables waste do not obtain the toxic heavy metals (such as Hg, Pb, Cd and As). All residue samples possessed suitable nutrient. Waste excellent biogas sludge used as natural fertilizer because it can be directly available for the plants and very good impact on the environment.

Acknowledgements

I would like to express my gratitude to Dr Aung Kyaw Thin, Rector, Banmaw University and Dr Aye Aye Han, Pro-rector, Banmaw University for their interest and encouragement on my research work. I also wish to express my thanks to Dr Myint Myint Sein, Professor and Head Department of Chemistry, Banmaw University for her kind help and invaluable guidance for this research work and providing with necessary facilities.

References

Börjesson, P.,and B.Mattiasson, (2007).Biogas as a resource-efficient vehicle fuel. Trends in Biotechnology, **2**(6)7-13

FAO, (2008)."Guide to Laboratory Establishment for Plant Nutrient Analysis".Italy:FAO, Rome, 30-44

- Hilkiah, M. F. N.,(2008), "Effect of Total Solids Concentration of Municipal Solid Waste on the Biogas Produced in an Anaerobic Continuous Digester". **4**(2) 21-33
- Johns, A.,(1998). "Agricultural use of Farm's wastes". 2ndEd Graw-Hill. (New York). 9(1), 67-77
- Nayono S. E., C. Gallert, J .Winter, (2009). "Food waste as a co-substrate in a fed-batch anaerobic biowaste digester for constant biogas supply". Water Science and Technology, **59**(6): 1169–1178

PANIDA,S.C., (2013). "Organic Farming" National Academy of Veterinary Sciences, (India). 3, 45-56

Characterization of Prepared Metakaolinite and Rice Husk Ash-based Geopolymer

Kyi Kyi San^{*}

Abstract

Geopolymer, an inorganic polymer, is a class of three- dimensionally networked alumino-silicate materials, similar to natural zeolite minerals. Metakaolinite and rice husk ash-based geopolymer has been prepared by using the different concentration of NaOH at 200°C for 4 hours. The source materials, kaolinite clay minerals from Kyauktaga and Taungnauk (Kyaukpadaung Township) and rice husk ash sample from Paddy Grinding Mill, Seik Phyu were collected. The collected samples were also characterized by using EDXRF, SEM, XRD, FT-IR and TG-DTA instrumental techniques according to the catalogue procedure. According to EDXRF analysis, the silica (SiO₂) and alumina (Al₂O₃) contents of Kyauktaga metakaolinite were found to be 57.773% and 35.756%, respectively. The silica (SiO₂) content (70.360%) and the alumina (Al₂O₃) content (23.022%) were found to be in Taungnauk metakaolinite. The mineralogical and microstuctural characteristics of geopolymeric materials were studied by modern instrumental techniques such as EDXRF, SEM, XRD, FT-IR, and TG-DTA. The silica (SiO_2) content in geopolymer sample was found to have highest percent (78.145%). Alumina (Al_2O_3) content was the second highest percent (16.175%) in geopolymer sample by EDXRF analysis. From the thermal analysis data, geopolymer sample was found to be the weight loss (19.452%).

Keywords: metakaolinite, rice-husk ash, geopolymer, alumino-silicate materials, instrumental techniques

Introduction

Geopolymer technology has emerged as an effective solution for the stabilization of industrial by-products and the immobilization of heavy metals. It has been established that the nature of the source materials used during synthesis has a significant effect on the final chemical and physical properties of the geopolymeric matrix (Van Deventer, 2004).

Geopolymerization involves a chemical reaction between solid alumino-silicate oxides and alkali metal silicate solutions under highly alkaline conditions yielding amorphous to semi-crystalline three dimensional polymeric structures, which consist of Si-O-Al bonds (Ravindra and Ghosh, 2009).

Unlike conventional organic polymers, glass, ceramic, or cement, geopolymers are non-combustible, heat resistant, formed at low temperatures, and fire and acid resistant (Wang and Cheng, 2003). It was known that a great number of minerals-industrial wastes were studied as raw materials for geopolymer synthesis, including pozzolana, natural aluminosilicate minerals, MK, fly ash, granulated blast furnace slag, fly ash and kaolinite mixture, red mud and MK mixture (Lopex, 2014).

^{*} Dr, Associate Professor, Department of Chemistry, Banmaw University

Adsorption and ion exchange processes in water pollution control are with the potential uses of the organic and inorganic polymers. Therefore, in the approach to replace the conventional adsorbents, geopolymers are a new strategy for decontamination of metal ions from waste water, which are composed of silica and alumina similar to zeolite material. For this reason, the geopolymers has begun to develop as adsorbent materials in the process of removal metal ions from the wastewater as an alternative to the industrial sector (Van Deventer, *et al.*, 2007).

Generally, the formula of the geopolymers is $M_n(-(Si-O_2)_z-Al-O)_n.wH_2O$, where M is an alkaline cation (Na, K or Ca), z is generally assigned a value of 1, 2 or 3 and n is the degree of polymerization and w is the number of water molecules (Van Jaarsveld, *et al.*, 2002). Geopolymers are similar to zeolites in chemical composition, but they reveal an amorphous microstructure. Geopolymer has the potential to reduce greenhouse emissions from the cement industry by 80% (Xu and Deventer, 2000).

Geopolymers are a novel class of materials that are formed by the polymerization of silicon, oxygen, and aluminium species to form an amorphous three-dimensional framework structure. These materials are sometimes referred to as alkaline cements or geocements and the overall technology, which encompasses the polymerization of inorganic metal species, as inorganic polymer technology (Krivenko, 1994).

The utilization of pure materials, including clays such as metakaolinite, for the formation of geopolymeric materials has been the subject of research undertaken by Joseph Davidovits since the early 1970s (Davidovits, 1979).

In this research work, the characterization of local metakaolinite and rice husk ash were carried out. The composition and microstructure of prepared geopolymers were investigated. The mineralogical and microstructural characteristics of geopolymeric materials were studied by means of EDXRF, SEM, XRD, FTIR and TG-DTA.

Materials and Methods

All the chemicals used were obtained from reagent grade (BDH). It comprised of sodium hydroxide, sodium silicate, 10% sulphuric acid and10% nitric acid. The apparatus used consist of Balance (Sartorius AG Gottingen BL 2105, L. Switzerland (210 g \pm 0.1 mg), Oven (Ambient 250 \pm 2 °C, Griffin, England), Furnace (100 – 1100 °C) (Model -N -No.9511028, Hohr-1995, Naberthem, Germany), pH Meter(Jenway 4330, Labquip,England), X-ray Diffractometer (XRD-Righaku-D-Max-2200, Japan), Energy Dispersive-X-ray Fluorescence Spectrometer (EDX-700, Shimadzu,Japan), SEM (Jeol – JSM – 5610 LV model, Japan), FT-IR (Perkin Elmer 1600 Fourier Transform Infrared Spectrometer, Japan) and TG-DTA (Hi-TGA 2950 Thermo- gravimetric Analyzer).

Sample collection

Kaolinite

Both kaolinite samples were collected from Chauk's Ceramic Bulb Industry. Representative kaolinite samples produced from (1) Kyauktaga (KTG) Area and (2) Taungnauk (TN) Area, near Popa Mountain, Kyaukpadaung Township, Mandalay Region. The beneficiated samples were bagged in cotton container. Sampling was carried out by cone and quartering method. The photographs of kaolinite powered samples are as shown in Figure 1 (a) and (b).



Figure 1. Kaolinite samples (a) Kyauktaga (b) Taungnauk

Metakaolinite

Metakaolinite was carried out by dehydroxylation of kaolinite at 700 °C for 20 hours. The dried sample was crushed by using the mechanical agate motor, and was sieved through the 200 mesh. Finally, the powdered metakaolinite sample was weighed and stored in a sealed plastic container. The photographs of metakaolinite powered samples are as shown in Figure 2 (a) and (b).



Figure 2. Metakaolinite samples (a)Kyauktaga (b)Taungnauk

Rice husk and rice husk ash

Representative rice husk samples were procured from Paddy Grinding Mill, which is located at Seik Phyu, Magway Region. The paddy hush was leached with HCl at 5M for one night. After leaching, the husk was thoroughly washed with distilled water and then dried. Then, about 30g of the acid-treated husk sample were taken in alumina crucible, placed in an electric muffle furnace and burned at 700°C for 4 h. The photograph of rice husk sample and white ash obtained after the thermal treatment is as shown in Figure 3 (a) and (b).



Figure 3. (a) Rice husk sample (b) Rice husk ash sample

Preparation of metakaolinite and rice husk ash-based geopolymer

Different ratios of metakaolinite and rice husk ash (total mass 9g) were mixed with the different concentrations of sodium hydroxide solutions and the same concentrations of sodium silicate solution (various volume ratios) on a non-absorbent base for various geopolymers. The specimen was removed from the mould after 30 min, and then prepared geopolymer specimens were dried at 200°C for 4 hours in oven. The photograph of prepared geopolymer sample is shown in Figure 4.



Figure 4. Metakaolinite and rice husk ash -based prepared geopolymer sample

Characterization of metakaolinite and rice husk ash samples

The powdered metakaolinite and rice husk ash samples were subjected to characterize by EDXRF, SEM, XRD, FTIR and TG-DTA techniques.

EDXRF measurement

Metakaolinite and rice husk ash samples were semi-quantitatively analyzed by EDXRF (Energy Dispersive X-ray Fluorescence) spectrometer. The data processing was carried out by using direct comparison standard method. The characteristic features of EDXRF spectra of metakaolinite and rice husk ash samples are shown in Figure 5 (a), (b) and (c), respectively. According to EDXRF analysis, the elemental compositions of metakaolinite and rice husk ash samples are presented in Table 1.

Surface analysis by SEM

Metakaolinite and rice husk ash samples were analyzed by Scanning Electron Microscope (SEM) with operation voltage of 10-15 KV and bean current of 50μ A for a visual inspection of external porosity and micro-texture. SEM photomicrographs of the metakaolinite and rice husk ash samples are shown in Figure 6 (a), (b) and (c) respectively.

XRD measurement

Metakaolinite and rice husk ash samples were qualitatively analyzed by X-ray diffractometer, Rigaku-D-max 2200, Japan. The resultant XRD spectra of metakaolinite and rice husk ash samples are shown in Figure 7 (a), (b) and (c) respectively.

FT-IR analysis

Metakaolinite (Kyauktaga and Taungnauk) and rice husk ash (Seik Phyu) samples were analyzed by IR Spectrophotometer. The procedure was used in accordance with the recommended standard procedure as reported in the FTIR Spectrophotometer catalogue. The characteristic features of FTIR spectra of metakaolinite and rice husk ash samples are shown in Figure 8 (a), (b) and (b). The spectral assignments for metakaolinite and rice husk ash samples are also presented in Table 2.

Thermal analysis of metakaolinite by TG-DTA

Metakaolinite samples were analyzed by thermo gravimetric differential analysis (TG-DTA). The procedure was followed according to catalogue (STA-449C, NEF 25 CH). According to TG profile of metakaolinite samples, there were only two breaks within the range of temperature, i.e., 600 °C. The onset temperatures show the loss in weight of water, i.e., surface water and phase change. The DTA profile indicates the exothermic peak (combustion) and the endothermic peak (slow burning and dehydration). The resultant thermograms of metakaolinite are shown in Figure 9 (a) and (b). The thermal analysis data of metakaolinite is also presented in Table 3.

Characterization of prepared geopolymer sample

The powdered metakaolinite and rice husk ash –based prepared geopolymer sample was subjected to characterize by EDXRF, SEM, XRD, FT-IR and TG-DTA techniques.

Results and Discussion

Characterization of metakaolinite and rice husk ash

Metakaolinite clay minerals and rice husk ash samples were semi- quantitatively analyzed by EDXRF spectrometer and the data are given in Table 1. The EDXRF spectra of metakaolinite from different localities and rice husk ash are shown in Figure 5 (a), (b) and (c), respectively. From Table 1, it can be found that silica was present in all samples. These samples were also qualitatively characterized by X-ray diffractometer.

SEM was also analyzed to describe the morphology of metakaolinite rice husk ash. The SEM photomicrographs of metakaolinite presented in Figure 6 (a) and (b) were observed as crystalline nature. From the scanning electron micrographs of rice husk ash Figure 6 (c), it can be clearly seen that the crystalline and amorphous nature of rice husk ash sample was observed in SEM image. From surface morphology of rice husk ash sample, many small pores and rod shape particles with diameter < 10 μ m were seen on the surfaces of rice husk ash sample.

From the XRD diffractograms, SiO_2 (silica) and Al_2O_3 (alumina) were observed in both clay minerals and rice husk ash samples as shown in Figure 7 (a), (b) and (b).

From the FTIR band assignments of Kyauktaga metakaolinite sample, the bands at 1087.89 cm⁻¹ (Si-O stretching vibration), 794.70 cm⁻¹ (Al-O stretching vibration),

621.10 cm⁻¹ (Si-O bending vibration) and 478.36 cm⁻¹ (Si-O in-plane bending vibration) were observed in Kyauktaga metakaolinite sample. FT-IR measurement was carried out on Taungnauk metakaolinite sample. The band assignments of metakaolinite and rice husk ash samples are tabulated in Table 2. According to FT-IR data, the bands at 1041.60 cm⁻¹ (Si-O stretching vibration), 794.70cm⁻¹ (Al-O stretching vibration), 699.40cm⁻¹ (Si-O bending vibration), 559.38 cm⁻¹ (Si-O-Al bending vibration) and 474.50 cm⁻¹ (Si-O in-plane bending vibration) were observed in Taungnauk metakaolinite sample. According to FT-IR data, the bands at 1099.46 cm⁻¹ (Si-O stretching vibration), 796.63 cm⁻¹ (Al-O stretching vibration), and 470.65 cm⁻¹ (Si-O in-plane bending vibration) were observed in rice husk ash sample. The FT-IR spectra of metakaolinite and rice husk ash samples are shown in Figure 8 (a), (b) and (c).

In this research, TG-DTA measurements on the collected local metakaolinite samples (Kyauktaga and Taungnauk) were carried out. TG-DTA thermograms of metakaolinite samples were shown in Figure 9 (a) and (b). From these observations, metakaolinite samples were found to be the decomposition processes. It was found the weight losses with respect to temperature. Heating temperatures were changed from (39-600) $^{\circ}$ C. According to TG-DTA curves, the maximum weight loss percent of these samples were observed as presented in Table 3. It had been found that the weight loss 0.03% for Kyauktaga metakaolinite sample and 0.82% for Taungnauk metakaolinite sample.



Figure 5. EDXRF spectra of metakaolinite and rice husk ash samples

Element as Oxide	Relative Abundance (%)(MK) (KTG)	Relative Abundance (%)(MK) (TN)	Relative Abundance (%) (RHA)
SiO ₂	57.773	70.360	98.472
Al ₂ O ₃	35.756	23.022	-
Fe ₂ O ₃	2.047	2.512	0.405
SO ₃	2.262	0.613	-
K ₂ O	1.064	2.550	-
TiO ₂	0.758	0.616	0.126
CaO	0.176	0.198	0.888
SrO	0.067	0.006	-
V ₂ O ₅	0.035	0.023	-
ZrO ₂	0.021	0.021	-
As ₂ O ₃	0.016	-	-
Ir ₂ O ₃	0.008	-	-
MnO	0.006	0.023	0.082
CuO	0.005	0.005	0.012
Cr ₂ O ₃	0.004	0.019	-
ZnO	0.002	0.005	-
Rb ₂ O	-	0.013	-
Ag ₂ O	-	0.006	-
NiO	-	-	0.016

Table 1Relative Abundance of Metal Oxides in Metakaolinite and Rice Husk Ash
Determined by EDXRF Analysis



Figure 6. SEM images of metakaolinite and rice husk ash samples



Figure 7. X-ray diffractograms of metakaolinite and rice husk ash samples



Figure 8. FT- IR spectra of metakaolinite and rice husk ash samples



Figure 9. TG-DTA thermograms of metakaolinite samples

(a) Kyauktaga (b)Taungnauk

Table 2FT-IR Data of Metakaolinite Samples (Kyauktaga and
Taungnauk)andRice Husk Ash sample

	Observed Wave	Observed Wave	Observed Wave	Band Assignment
No.	No. (cm ⁻¹)	No. (cm ⁻¹)	No.(cm ⁻¹)	(Functional Group)
	(MK) (KTG)	(MK) (TN)	(RHA)	
1	3454.62	3421.83	-	O-H stretching vibration
2	1631.83	1641.48	-	O-H bending vibration
3	1087.89	1041.60	1099.46	Si-O stretching vibration
4	794.70	794.70	796.63	Al-O stretching vibration
5	621.10	699.40	-	Si-O bending vibration
6	-	559.38	-	Si-O-Al bending vibration
7	4878.36	474.50	470.65	Si-O in-plane bending vibration

Metakaolinite Sample	Temperature Range (°C)	Weight Loss (%)	Remark
Kyauktaga	39.46-600.26	0.03	Dehydration due to surface and absorbed water
Taungnauk	39.46-600.26	0.82	Dehydration due to surface and absorbed water

Table 3Thermal Analysis (TG and DTA) Data of Metakaolinite Samples

Characterization of prepared geopolymer sample

Determination by EDXRF spectrometry

Relative quantitative elemental determination of prepared geopolymer sample was analyzed by using EDXRF technique and the data was given in Table 4. The EDXRF spectrum of prepared geopolymer sample is shown in Figure 10. From EDXRF measurement, the highest percent (78.145%) of silica content had been found that in this prepared geopolymer sample.



Figure 10. EDXRF spectrum of prepared geopolymer sample

Table 4 Relative Abundance of the Prepared				
Geopolymer	Geopolymer Sample by EDXRF			
Analysis (m	ass %)			
Element as Oxide	Relative Abundance (%)			
SiO ₂	78.145			
Al ₂ O ₃	16.175			
Fe ₂ O ₃	2.098			
K ₂ O	1.948			
SO ₃	0.714			
TiO ₂	0.489			
CaO	0.331			
Cr ₂ O ₃	0.022			
MnO	0.021			
V ₂ O ₅	0.019			
Rb ₂ O	0.013			
CuO	0.008			
ZnO	0.007			
SrO	0.006			
Y2O ₃	0.003			

Scanning electron microscope

The Scanning Electron Micrograph of prepared geopolymer sample was obtained at 200°C for 4 hours. SEM was performed on this geopolymer sample to investigate the microstructure. From micrograph shown in Figure 11 (a) and (b), it had been observed that the microstructure of prepared geopolymer sample by increasing the silica and alumina contents, the morphology changes and a more integrated phase occurred.



Figure 11. SEM image of prepared geopolymer sample

X-Ray Diffraction

X-ray powder diffraction patterns of metakaolinite and rice husk ash –based geopolymer sample with NaOH concentration (8M) at 200°C is shown in Figure 12. The X-ray diffractogram of the prepared geopolymer sample was taken to confirm its structure, purity and crystallinity. XRD analysis was carried out to identify new formed phases and the degree of amorphicity.

FT-IR Spectrum analysis

The FT-IR spectrum of metakaolinite and rice husk ash-based prepared geopolymer sample is shown in Figure 13. During geopolymerization, the prominent band at 1041.60 cm⁻¹ presented in IR spectrum of metakaolinite shifts towards a lower wave number. The corresponding IR band of this prepared geopolymer sample was 1020.38 cm⁻¹. According to Table 5, it had been observed that Si-O stretching vibrations in original rice husk ash was 1099.46cm⁻¹, while it was shifted to lower values in geopolymer sample around 1020.38 cm⁻¹ in IR spectrum of prepared geopolymer sample.



Tunes of	F Wave Number (cm ⁻¹)						
Species	Metakaolinite (TN)	Rice Husk Ash	Geopolymer sample	Band Assignment			
Si -O	1041.60	1099.46	1020.38	stretching vibration			
Al -O	794.70	796.63	775.81	stretching vibration			
Si -O	699.40	-	-	symmetric stretching vibration			
Si -O	474.50	470.65	451.36	in-plane bending vibration			

Table 5IR Bands and their Corresponding Species of Prepared Geopolymer
Sample

On the aspect of TG - DTA of prepared geopolymer sample

Thermal stability of metakaolinite and rice husk ash-based prepared geopolymer was investigated by TG-DTA analysis. The TG-DTA thermogram of prepared geopolymer sample was shown in Figure 14. The results were summarized in Table 6 for prepared geopolymer sample. From this thermogram, the endothermic peak at about 69.78°C probably observed geopolymer decomposition in prepared sample. The geopolymer decomposed with a sharp loss in weight (19.452 %) was observed in prepared geopolymer sample.



Figure 14. TG-DTA thermogram of prepared geopolymer sample

Table 6	Decomposition	Temperature	and	Weight	Loss	(%)	of	Prepared
	Geopolymer San	nple						

Geopo	TG temp-	Weight loss	TG	Peak	DTA
-lymer	erature	(% w/w)	remark	Temp	Remark
	range (°C)	between Temp break		(°C)	
Sample	36.94 - 601.52	19.452 %	Probably due to unburned carbon	69.78 (Endo peak)	Dehydration of surface and absorbed water

Conclusion

In this investigation, collected kaolinite samples from Kyauktaga andTaungnauk, Kyaukpadaung Township, Mandalay Region were analyzed and characterized by modern instrumental techniques.

From the results of EDXRF analysis, SiO_2 content in rice husk ash was found to be the highest percent (98.472%). From XRD diffractogram, SiO_2 (silica) was observed in rice husk ash sample. It can be concluded that the compositions of metakaolinite (Kyauktaga and Taungnauk) and rice husk ash (Seik Phyu) were found to be used as geopolymer.

From SEM micrographs, metakaolinite samples were observed as crystalline nature. Moreover, SiO₂ content in metakaolinite samples were very similar and contained the highest content in both samples. The 2θ peaks in the XRD patterns of collected metakaolinite samples were well matched with standard peaks of metakaolinite, silica and alumina. From the XRD diffractograms, SiO₂ (silica) and Al₂O₃ (alumina) were observed in both metakaolinite samples.

FT-IR data indicated the presence of Si-O and Al-O functional group in metakaolinite sample. TG-DTA curves indicated the maximum weight loss percent of (0.03%) in Kyauktaga metakaolinite, whereas the minimum weight loss percent of (0.82%) in Taungnauk metakaolinite. Two processes were found in TG-DTA thermograms; first process was dehydration of absorbed water in the clay samples and second was decomposition of hydroxide groups in the clay structure.

Characterization and identification were also done by modern instrumental techniques for metakaolinite and rice husk ash-based prepared geopolymer sample. Prepared geopolymer sample was characterized by EDXRF, SEM, XRD, FT-IR and TG-DTA.

According to EDXRF results, silica (SiO₂) content in prepared geopolymer sample was found to have highest percent (78.145%). Alumina (Al_2O_3) content was the second highest percent (16.175%) in geopolymer sample. According to SEM analysis, the microstructure of prepared geopolymer sample may be viewed as composites comprising of aluminosilicate gel phase. XRD analysis was carried out to identify new formed phases and the degree of amorphicity.

The most characteristic difference observed between FTIR spectra of metakaolinite, rice husk ash and geopolymer sample were shifted of the band attributed to the symmetric vibrations of Si-O-Al bond. This band, which appeared as a broad band at around 1041.60 cm⁻¹ in the FTIR spectrum of metakaolinite, became sharper and shifted to lower frequencies 1020.38cm⁻¹ in FTIR spectrum of geopolymer sample indicating the formation of a new product (the amorphous aluminosilicate) that is associated with dissolution of rice husk ash amorphous phase in the strong alkaline activating solutions. From thermal analysis data, it was found that the weight loss % of (19.452%) in prepared geopolymer sample. The temperature about 600°C was full decomposition of prepared geopolymer sample with weight loss per minute being detected in this sample.

Acknowledgements

The author would like to thank the Department of Higher Education, Mandalay, Myanmar, for allowing to carry out this research program. Profound gratitude is specially expressed to Ministry of Education, Myanmar. I wish to express my gratitude and deep appreciation to Dr Aung Kyaw Thin, Rector, Banmaw University, for invaluable guidance and constant encouragement. I would like to express my sincere gratitude to Dr Aye Aye Han, Pro-rector, Banmaw University, for giving me permission to conduct this research and providing with necessary facilities. I would like to express my gratitude and deep appreciation to Professor Dr Myint Myint Sein, Department of Chemistry, Banmaw University, for her suggestion, invaluable guidance and encouragement while doing my research work.

References

- Davidovits, J. (1979). "Synthesis of New High-Temperature Geo-polymers for Reinforced Plastics/ Composites", *In Proceedings of PACTEC '79*, Society of Plastic Engineers, 151-154.
- Krivenko, P. V. (1994). "Alkaline cements, In 1st International Conference on Alkaline Cements and Concretes", 1, 11-130
- Lopex, G.F.J. (2014). "Stuidy of Geopolymer Adsorbents Prepared from Metakaolin and Rice Husk Silica for Targeting to Heavy Metal Capture", Energy and Environment Science, Japan, 1-105
- Ravindra,N.T., and S.,Ghosh. (2009). "Effect of Mix Composition on Compressive strength and Microstructure of Fly Ash -based Geopolymer Composites", Asian Research Publishing Network Journal of Engineering and Apply Sciences, India, 4, 68-74
- Van Deventer, J.S.J., Jaarsveld, J.G.S., and G.C. Lukey. (2004). "A Comparative Study of Kaolinite Versus Metakaolinite in Fly Ash based Geopolymers Containing Immobilized Metals", Department of Chemical and Biomolecular Engineering, University of Melbourne, Victoria, Australia, 191, 531-549
- Van Deventer, J.S.J., P., Duxon, and G.C. Lukey.(2007). "Reaction Mechanisms in the Geopolymeric Conversion of Inorganic Waste to Useful Products", *Journal of Hazardous Materials*, Australia; 139, 506-513
- Van Jaarsveld, J.G.S., J.S.J. Van Deventer, and G.C. Luke. (2002). "The Effect of Composition and Temperature on the Properties of Fly Ash and Kaolinite –based Geopolymers", *Chemical Engineering journal*, Department of Chemical Engineering, The University of Melbourne, Australia, 89, 63-73
- Wang, J.W., and T.W. Cheng. (2003). "Production Geopolymer Materials by Coal Fly Ash", Proceeding of the 7th International Symposium on East Asian Resources Recycling Technology, National Taipei University, Taiwan, 263-266
- Xu, H., and J.S.J.V. Deventer. (2000). "The Geopolymerization of Alumino-Silicate Minerals", International Journal of Mineral Processing, **59**, 247-266