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Chapter 3

METHANE PURIFICATION FROM LANDFILL USING PVC BASED MEMBRANE

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ABSTRACT

Mixed-Matrix Membrane is a developing technology that has been use in the gas separation process due to the ability of MMMs to cope with the limitation of polymeric membrane and inorganic membrane. Therefore, this research is conducting to study the permeability and selectivity of carbon dioxide (CO_2) and methane gas (CH_4) of polyvinylchloride (PVC) Mixed-Matrix-Membrane (MMMs) with the inorganic fillers of zeolite 4Å particles. The fabrication of MMMs is prepared by using dry/wet phase inversion method. Fourier Transform Infrared Spectroscopy (FTIR) is used to study the chemical interaction of the membrane by analyzing the intensity of the peak of chloride vibration. Meanwhile, Scanning Electron Microscope (SEM) is use to analyses the cross sectional morphology of MMMs. The performance of MMMs analyses by using Design of Expert

(DOE) method. While, the model regression equation is developed as the potential use for screening the permeability of CO_2 and CH_4 based on the effect of PVC and zeolite concentration.

Keywords: mixed-matrix membrane, selectivity of CO_2/CH_4, permeability of CO_2, permeability of CH_4

INTRODUCTION

Typically, landfill gas or biogas derived from a landfill comprises generally equally molal amounts of a mixture of carbon dioxide (CO_2) and methane (CH_4), with the CO_2 and CH_4 representing about 90 mole percent or more of the biogas. The landfill gas also contains minor amounts of nitrogen, oxygen, hydrogen, carbon monoxide and a variety of undesirable trade impurities present at the ppm level, as well as water vapour. The nitrogen and oxygen content of the biogas depends on the air ingress to the landfill- and gas-collection system. Elimination of contaminant CO_2 from natural gas and landfill gas streams, composed mostly of CH_4, is an important problem. The presence of CO_2 in natural gas significantly lowers the energy density of the gas stream and can lead to pipeline corrosion over time [1].

Biogas, specifically landfill gas, often contains too much CO_2 and too low a CH_4 concentration to fuel a natural gas engine for electrical power generation. CH4 gas stands as one of the most prevalent gaseous in the air. Global CH_4 emissions from landfill are estimated to be between 30 and 70 million tonnes each year [2]. CH_4 originating from landfill is vastly found in developed and populated countries, where the levels of waste tend to be the highest. CH_4's unique role as a greenhouse gas and as the primary component of natural gas means that reducing CH_4 emissions can yield significant economic, environmental and operational benefits. Companies are reducing their emissions of greenhouse gases, improving operational safety and enhancing the efficiency of their operations. Further economic and operational benefits can result when CH_4 mitigation activities reduce maintenance and fuel requirements or result in the capture of other valuable hydrocarbon resources. Currently in the market 1 m^3 of CH_4 is equivalent to $68.93 [3]. A recent study was carried out at Kampung Sg. Ikan Landfill in Kuala Terengganu, Terengganu, Malaysia. Municipal solid wastes from the city of Kuala Terengganu were gathered, weighed and datas were collected. Wastes were segregated according to their 13 types, such as 3D plastic, 2D plastic, glass, and so on (Table 1). CH_4 can be achieved or obtained through solid waste, mainly food waste, through the precise processes and through gas separation.

Among the many types of polymers that exhibit gas separation properties for gas mixtures, a few polymers such as polysulfone (PSf), polyethersulfone (PESf), polyetherimide (PEI) and polyimide (PI) have been recognized as promising polymers with respect to their permeability and selectivity. Nowadays, polymeric membranes dominate the industry because of the outstanding economy and competitive performance [7]. The membranes can be operated at ambient temperature and they have good mechanical and chemical properties [8]. PVC is a polymer with a wide variety of applications in different industries due to high compatibility with additives, easy process ability and recyclability. Despite the extensive use of PVC in several industrial applications, studies on the gas separation performance of PVC membranes are scarce [9]. Despite their suitability for various applications in research and commercialization, polymeric membranes are still ineffective in meeting the requirement for the current advanced membrane technology as these materials have demonstrated a trade-off between the permeability and selectivity, with an 'upper-bound' evident as proposed by Robeson [10].

To overcome the limitation in of polymeric membrane and inorganic membrane in gas separation, mixed-matrix membranes (MMMs) is develop as an alternative approach in gas separation process. MMMs are a hybrid membrane of organic-inorganic compounds that proposing better separation performance at reasonable price. The combination of both membranes gives a pleasant stability of molecular sieving and better performance of organic membrane [11]. The MMMs characterized by embedding inorganic materials into the polymeric matrix which can be any polymeric materials such as polysulfone, poly-vinyl-hloride and polyamides. Since MMMs are design to improve the host membrane of polymeric membrane, the selection of inorganic material is important due to the advantages of peculiar properties of inorganic fillers such as zeolite [12]. Therefore, the main factor in the fabrication of MMMs is the affinity between the two phases involved and the compatibility of hybrid organic-inorganic membrane.

Over the past decade, researcher use zeolite as an inorganic filler in the fabrication of MMMs because the characteristics of zeolite in having well-defined size, uniform pore distribution, high specific area and high prorsity [11]. Nonetheless, in phase separation, the critical issue found in the development of MMMs is the poor compatibility and adhesion of zeolite-polymer. In other hand, the fabrication of MMMs with zeolite based is highly cost since the preparation on the modification of zeolite is difficult in large-scale production. Therefore, due to the issues, a study is conducting to develop a newly combination of zeolite- PVC in the fabrication of MMMS.

Table 1. Municipal solid wastes data from Kg. Sg. Ikan landfill for 3 weeks

Categories/Week	1	2	3	Total
Total waste (kg)	11010.00	13500.00	7040.00	31550.00
Waste weighed (kg)	9926.95	12453.20	7007.90	29388.05
Waste weighed (%)	90.16	92.25	99.54	93.15
Plastic 3D (kg)	362.59	480.00	274.30	1116.89
Plastic 2D (kg)	869.10	1164.50	665.30	2698.90
Metal (kg)	7.96	33.70	33.20	74.86
Aluminum can (kg)	100.61	195.90	107.30	403.81
Paper (kg)	843.60	878.40	741.30	2463.30
Pampers (kg)	873.35	1631.00	889.40	3393.75
Glass (kg)	200.72	235.70	142.10	578.52
Wood/Landscape (kg)	1099.51	2179.20	1802.30	5081.01
Polystrene (kg)	118.90	219.70	81.10	419.70
Bed (kg)	9.48	0.00	10.00	19.48
Textile (kg)	108.94	289.50	146.40	544.84
Food waste (kg)	4542.91	5227.60	2107.60	11878.11
Others (kg)	1063.62	1046.80	66.50	2176.92

METHODS

Experimental

Chemicals

Polyvinyl chloride (PVC) is a polymeric material which grouped as a glassy polymer that will be use in the fabrication of membrane, which have been purchased from Sigma Aldrich. The properties of PVC in having high operating temperature enable the gas separation process work at maximum condition. N-Methyl-2-pyrrolidone (NMP) is use as a solvent in sample preparation, NMP which has chemical formula of C_5H_9NO and the average molecular weight of 99.13g/mol with 99.5% [13]. NMP is purchased from Sigma Aldrich. The molecular sieve involved in the preparation of Mixed-Matrix-Membranes (MMMs) is zeolite 4Å. Type 4Å is the sodium form of molecular sieve which it will absorb those molecules having a critical diameter of less than 4Å. The molecular sieve has particle size of 8 - 12 mesh purchased from Aldrich. Methane gas and Carbon dioxide gas with purity 99% were obtained from Air Products Malaysia Sdn. Bhd for gas permeation.

Sample Preparation

The approach method for the preparation of mixed-matrix membrane is through dry/wet phase inversion. The zeolite is prepared from 5 to 10 W/V% and dissolve it in varied volume of NMP with 90cm^3 to 95cm^3. Then, the mixture will mix homogeneously by stirring the mixture for three hours at condition of 200 rpm and 100°C. Weight the PVC with 5 to 10 W/V% and add the PVC to the solution until it reaches homogeneous mixture. In order to achieve yield homogeneous mixture, agitate the mixture at 300 rpm by using magnetic stirrer at the room temperature for another three hours. Store the dope solution at storage vessel and degasses all the bubble by leaving the dope solution overnight at room temperature. Prepare glass plate for the casting method by using glass rod. Finally, the sample membrane was ready for the permeation test for calculate the permeability and selectivity of CO_2 and CH_4 separation.

Single Gas Permeation Test for CO_2/CH_4 Separation

In this study the membrane use for each Single Gas Permeation Test has a diameter approximately 6cm. First, purified CH_4 at two bars was used as a gas test and connect the membrane with the gas permeation apparatus manufactured by Aba Manufacturing Sdn Bhd. Then, set the temperature at room temperature (25°C ± 5°C) and finally, measure the gas permeation rate by using soap-bubble meter manufactured by Dwyer. Repeat the procedures of Single Gas Permeation Test by replacing the test gas with CO_2 and set the pressure at one bar. Solubility of the pure gas is a factor that will affect the rate permeation gas. Therefore, CO_2 will be the last gas to be measure due to the encouragement of membrane plasticization.

Screening Study of Membrane for CO_2/CH_4 Separation

The experimental data is screening by using Design of Experiment (DOE) software, version 7.1. DOE is software that enables the users to interpret multi-factor experiments. This software promotes a wide range of design including factorials, fractional factorials and composite design. Meanwhile, in analysing an experiment DOE fit the model that relating with the response or quality characteristics to a set of controllable variables. In this study there are three factors were selected which are concentration of zeolite (X1), concentration of PVC (X2) and duration time of immersion for glass plate in water (X3) and selectivity of CO_2 and CH_4 as response which coded as Y in the software. The selectivity of $CO_2$and CH_4 that diffuse through the membrane analyse as response which denote as Y in the software. Each response parameter that containing notable term will be develop in mathematical model by using multiple linear regression analysis (MLRA) and analysis of variance (ANOVA).

RESULT AND DISCUSSION

Gas Performance Analysis

Single gas permeation test is used to study the permeability and selectivity of CO_2 and CH_4 for all samples of fabricated membrane. Three samples from each different eight composition of formulated membrane with vary concentration of zeolite, PVC and NMP were tested to get the average reading of result. The complete experiment for each of the sample and output response were tabulated at Table 2. The proportion differentiation between the whole design matrixes is less than 5% for all of the three samples of membrane. The range of the selectivity of CO_2/CH_4 is between 2.2777 to 2.8377. The condition of the single gas permeation test is 1bar. At low operating pressure the solubility coefficient of CO_2 was greater which promoted more permeation rate of CO_2 over polymeric dense membrane. Therefore, at 1bar single gas permeation test is most favourable method to test the permeability and selectivity of CO_2 and CH_4.

Table 2. Experimental design matrix and response results

Specimen	Concentration Variable			Experimental result
	X_1 (W/V %)	X_2 (W/V %)	Time, t (min)	Y
1	5	5	5	2.6039
2	10	5	5	2.6638
3	5	10	5	2.9539
4	10	10	5	2.6254
5	5	5	15	2.4281
6	10	5	15	2.6247
7	5	10	15	2.2777
8	10	10	15	2.8377

X_1, Concentration of zeolite; X_2, Concentration of PVC; and duration time of immersion for glass plate in water and Y, selectivity of CO_2/CH_4

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Fourier Transform Infrared Spectroscopy (FTIR) spectra is using to detect the presence of chemical added to the MMMs and the interactions between PVC and zeolite particles [14, 15, 16]. Figure 1 show characteristics for the sample of fabricated membrane with the highest loading of zeolite (10% W/V%) correspond with 5 W/V% of PVC of 15 min duration time for immersion of glass plate due the performance of the fabricated membrane obeys the upper bound Robeson plot. The selectivity of CO_2/CH_4 of fabricated membrane is 2.6247 with respect to the significant morphology for polymerization of membrane. From Figure 1, the peak value 3640 cm^{-1} is corresponding to the O-H bound in zeolite. The interaction shows that the bound between polymer and zeolite exists in the sample of fabricated membrane. However, the elimination of void spaces between the polymer matrix and zeolite are not enough through this interaction. Aromatic carbon double bond, C=O bare associated with the peak number is 1563cm^{-1} which indicates the aromatic bonds were not disrupted and remained in the membrane. Meanwhile, the band located at 2877 cm^{-1} was associated with CH_3 stretch vibration of the membrane. Meanwhile, the band located at 2877 cm^{-1} anhydride C-O stretch in the PVC/Zeolite mixed-matric membrane.

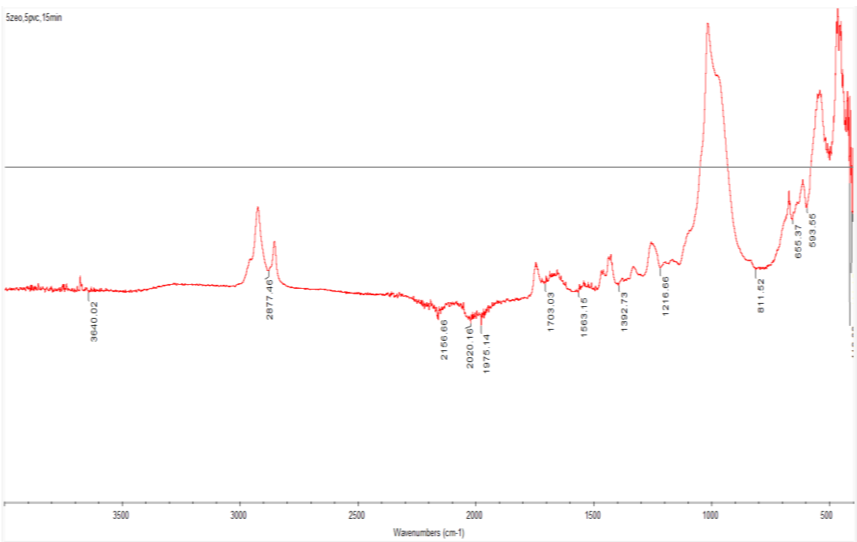


Figure 1. FTIR spectra of PVC/Zeolite mixed-matrix membrane.

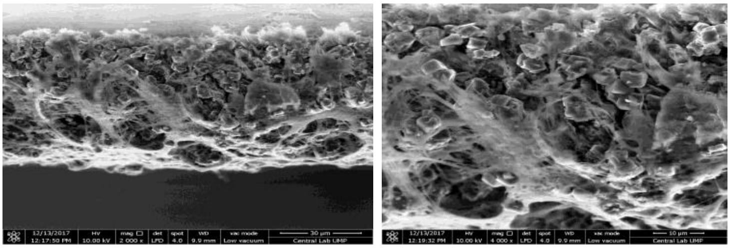
Table 3 demonstrated the summary for the characteristics of adsorption for the sample with the selectivity of CO_2/CH_4.

Table 3. Functional group of membrane sample with selectivity of CO_2/CH_4 in respect to the comparison of characteristics adsorption

Functional Group	Sample	Characteristics Adsorption
CH_3 stretch	2877.46	2865-2885
Aromatic carbon double bond C=O	1563.15	1600 or 1475
Anhydride C-O stretch	1216.66	1300
O-H bound in zeolite	3640	3200-3600

Scanning Electron Microscope (SEM)

SEM is used to study the morphology of the cross-sectional area of fabricated membrane. Figure 2 shows the cross-sectional area SEM image of PVC/Zeolite membrane taken from sample for 15 min duration time of immersion rate of 10 W/V% zeolite, 5 W/V% PVC. The Figure 2(a) show the SEM image of PVC/Zeolite MMMs with 2000X magnified while Figure 2(b) is more clear SEM image for the morphology of zeolite molecules. The presence zeolite molecules as a filler in polymeric membrane increases the adhesion of the polymer chain. Zeolite is an inorganic filler which enhance the flexibility of the polymeric membrane by improving the interaction between the heterogeneous phases of the matrix membrane. Furthermore, the adhesion of organic-inorganic phase interaction is improved with the presence of thin layer around of chemical composition the zeolite molecules as a chemical treatment for the matrix membrane.



(a) (b)

Figure 2. SEM images of the Mixed-matrix membrane of Zeolite/PVC; (a) 2000x magnified SEM image of PVC/Zeolite MMMs (b) 400x magnified SEM image of PVC/Zeolite MMMs.

ANOVA Analysis

The manipulated data of the study is expressed with the variable of concentration for Zeolite (X1), amount of Polyvinylchloride (X2), and time taken for immersion of membrane (X3) with correspond output of selectivity of CO_2/CH_4 (Y3). This research approach two statistical point of view to analysed and asses the model of the experimental data by using significance of factor (SOF) and R-squared (R^2) test show the result for SOF and the interaction for the selectivity of CO_2/CH_4 with the value of R^2 for the analysed model is $R^2>0.7$ which is the variation of the response could be justified by using mathematical modelling. From Table 4, the original value of R^2, adjusted R^2 and predicted R^2 are demonstrated after neglecting the insignificance terms of the design model. The predicted R^2 is 0.948237 is in reasonable agreement with adjusted R^2 because the value is within 0.2. In other words, the design model of the experiment is in an accurate description of experimental data which indicated the relationship between the variables and response data. The model result shows the tendencies for the model to form linear regression fit which showed that the experimental research range is adequate.

Table 4. R^2 statistic for the fitted model

Model source	Selectivity of CO_2/CH_4
Std. Dev.	0.023085
Mean	2.530733
Coeddicient of variation	0.912191
R-Squared	0.996765
Adj R-Squared	0.988677
Pred R-Squared	0.948237
Adeq Precision	31.53732

Empirical Model Analysis

The approach model is well fitted to the case study (experimental result), since from Table 4 the value of $R^2 > 0.90$ which is 0.988677. In other words, the selectivity of CO_2/CH_4 is reliable to the regression model of the membrane's permeability. Meanwhile, Table 5 demonstrated the result of F-test ANOVA from the regression model with 95% confidence level. The model value of F-value is 123.241 and the prob > F is less than 0.0500 which implies that the model is significant. In other words, the improvement of the experimental model is achieved by discarding insignificant effects term after completing all the eights sample. Table 5 show the result of F-test ANOVA for the selectivity of CO_2/CH_4.

Table 5. ANOVA statistic for the fitted model

	Sum of Squares	DOF	Mean Square	F- Value	p-value Prob > F
Model	0.32839	5	0.06568	123.241	0.0081
A-X1: Concentration of Zeolite	0.19759	1	0.19759	370.776	0.0027
B-X2: Concentration of PVC	0.0195	1	0.0195	36.5852	0.0263
AB	0.06929	1	0.06929	130.0181	0.0076
AC	0.008182	1	0.008182	15.35316	0.0594
BC	0.033827	1	0.033827	63.47446	0.0154
Residual	0.001066	2	0.000533		

At 10% Zeolite loading (highest loading of zeolite) the void size is greater than the diameter of the gas molecules which enable the gas molecules to pass through (penetrate) the voids with lower diffusion resistance instead of selective pores of zeolite.

The interaction of A: concentration of zeolite and B: concentration of PVC (AB) have the most significant effect on the selectivity of CO_2/CH_4 with the F-value is 130.0181 and the P-value is only 0.0076% which is less than 0.05%. The interaction of zeolite as an inorganic membrane with PVC (polymeric membrane) is significant because of adhesion of hybrid membrane. During the casting process, the PVC is detached from the zeolite surface producing the micro-cavities throughout the membrane. However, functionalized molecular sieves are required to achieve high selectivity of CO_2/CH_4 and good performance of mixed-matrix membrane due to the great interaction between the polymeric membrane phase and sieves.

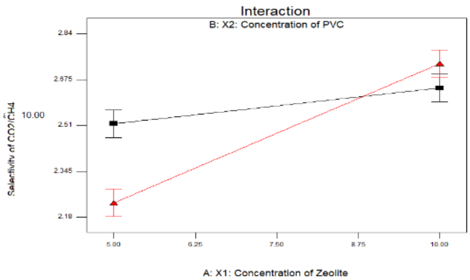
Meanwhile, the interaction of AC and BC where C: time duration for immersion of membrane in minute resulted is significant with the f-value are 15.3516 and 63.4766 respectively. While the p-value for AC interaction is 0.0594 which is slightly from 0.05% for p-value and BC interaction is 0.0154 which less than 0.05% to achieve significant data.

Verification on Statistical Models and Diagnostic Statistic

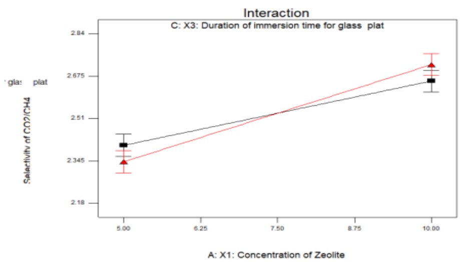
The interaction between independent factors in the experimental model is being investigated through Response surface method to observe the effects among the variables.

Figure 3 demonstrated the interaction between the independent variables by combining two independent factors in binary combination for all the responses. Figure 3(a) show that the interaction between A: Concentration of zeolite and B: Concentration on of PVC with the diagonal data obtained shows that as the increment of AB interaction increase the selectivity of CO_2/CH_4. Meanwhile, Figure 3(b) the interaction of AC demonstrated the same pattern of AB interaction where C: duration of immersion time of glass plate in minute. Meanwhile, the standard deviation of the experimental model is test through demonstrated plotted data of the normal probability of the residual as shown in Figure 4(a). The plotting data is important to ensure the actual and predicted response value is still obeying the normal distribution. The residual data versus predicted is plotted in Figure 4(b) with the data scattered randomly in constant range of residual across the graph within the horizontal line. It shows that the experimental model proposed is feasible and constant amount of variance is confirmed.

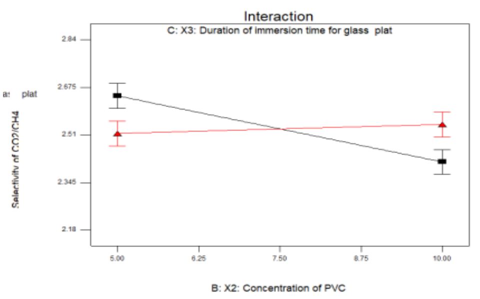
In other hands, the reliability of the empirical data is confirmed from the comparison of predicted output data from regression model with the actual values obtained from experimental results as shown in Figure 4(c). Generally, the predicted value is directly proportional to the actual value of the experimental results. In other words, the value of the predicted data increases as the increment of the selectivity of CO_2/CH_4 as well as the actual value of the experimental results. The output result from the experimental response is well fitted in acceptable variance range. Therefore, the regression model obtained from DOE is enabling to further as a predictor for the analysis of variability of membrane concentration to produce high selectivity of CO_2/CH_4.



(a)



(b)



(c)

Figure 3. Interaction via Selectivity of CO/CH for (a) concentration of PVC and zeolite (b) concentration of Zeolite and Duration of immersion time for glass plat
(c) Concentration of PVC and Duration of immersion time for glass plat.

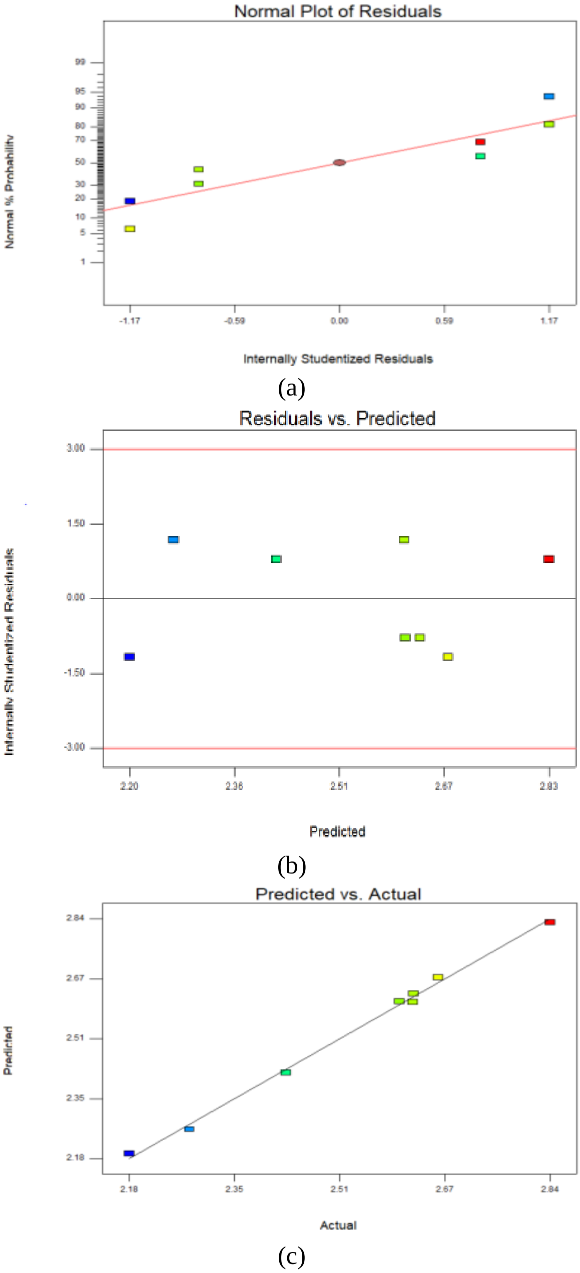


Figure 4. (a) Normal probability plot of residual; (b) Plot of residual versus predicted response and (c) Predicted vs. actual values plot.

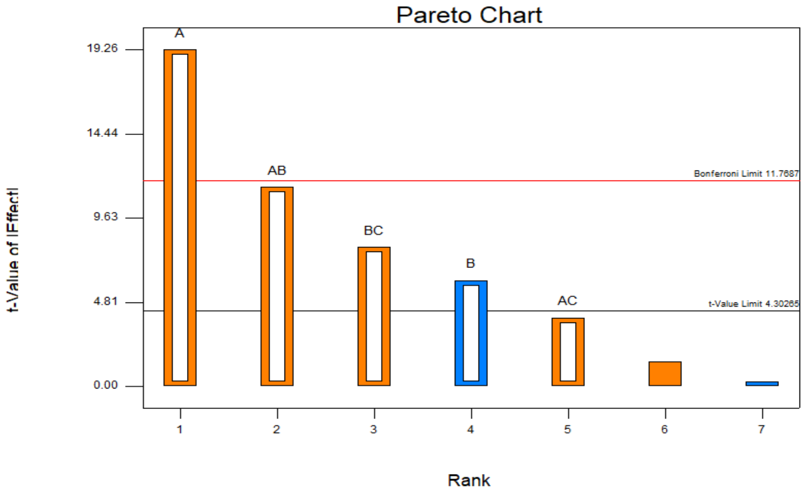


Figure 5. Pareto chart.
A: Concentration of Zeolite (W/V%)
B: Concentration of PVC (W/V%)
C: duration time of immersion for glass plate in water.

From Figure 5, the Pareto chart shows the f-value from the ANOVA which demonstrated that there is a variation on the value of effects on the difference type of factors of the model. The Pareto chart show that the factor of A is 19.26 which is the highest amongst the others and it at the first rank in Pareto chart. Meanwhile, the interaction between AB, BC and AC show the different f-value which are 11.7687, 1.64 and 4.30265 respectively. The factor B is insignificant in the Pareto chart as it exists in blue colour which ANOVA analysis indicated it as insignificant factor. The Bonferron limit is at 11. 7687 which the peak of AB interaction. While, the t-Value limit is at the peak of the interaction of AC.

Model Equations Based on Screening Effect

Figure 3 demonstrated the interaction for all the factors in the experimental model which showed that the membrane is performed in linear model. The factors of the model are in significant model term as well as the selectivity of CO_2/CH_4. The model term is A; Concentration of Zeolite (W/V%), B; Concentration of PVC (W/V%) an and C; Time taken for immersion of membrane in minute. From Table 5, the F-test ANOVA analysis performed the regression to assess the equation of the experimental model with 95% confidence level.

Equation 1 expressed the regression model equation and coefficient for selectivity of CO_2/CH_4.

$$\text{Selectivity of } CO_2/CH_4 = 2.53 + 0.16A - 0.049B + 0.093AB + 0.032AC + 0.065BC \quad (1)$$

where;

A: Concentration of zeolite

B: Concentration of PVC

C: Duration time of immersion for glass plate in water

CONCLUSION

The fabrication of Poly-Vinyl-chloride and zeolite are to investigate the effect of polymer concentration and additive of (zeolite) on the permeability of CO_2 and CH_4 as well as the selectivity of CO_2/CH_4. The homogeneous mixture of polymer and zeolite were miscible with each other. From FTIR result the existence of 3640 cm^{-1} peak explained the presence of of an interaction between polymer and zeolite. The highest selectivity of CO_2/CH_4 are observed to be 2.8377. meanwhile, the result from DOE expressed that these mixed-matrix membranes are affected with the interaction of concentration of zeolite, concentration of PVC and duration time for the immersion rate of glass plate to offer high selectivity of CO_2/CH_4 by using ANOVA analysis. The regression equation is developed from regression model is expressed in [Equation 1 for the selectivity of CO_2/CH_4 based on the effect of polymer concentration.

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