VOL. 12, NO. 20, OCTOBER 2017 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

DEVELOPMENT OF HYBRID COCONUT SHELL-PEEK ADSORBENT FOR METHANE ADSORPTION: OPTIMIZATION USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Adsorbed natural gas (ANG) provides efficient and clean combustion, with minimal emissions compared to diesel and gasoline. This article was designed to develop techniques of ANG for transportation application by apply RSM and CCD to identify the optimum preparation conditions for preparation of stable adsorbent for methane adsorption. Coconut shell and poly ether ketone (PEEK) was selected for synthesis of activated carbon (AC). The effectiveness of the parameters was determined using response surface method (RSM) couple with central composite design (CCD). The analysis of variance (ANOVA) was applied to identify the significant parameters. The quadratic model was adopted, as it has the highest F-value of 21.62 and P-value of less than 0.05, which relate the parameters and response. Microwave power has the highest F-value of 62.36. The maximum methane uptake of 5.12mmol g^{-1} was achieved. Overall, the hybrid coconut-PEEK adsorbent was found to be suitable for CH_4 adsorption.

Keywords: sustainable energy; adsorbed natural gas; methane; optimization; response surface methodology; central composite design.

1. INTRODUCTION

The global problem of natural gas vehicles (NGVs) is finding the best techniques of increasing the energy density of their usable fuel. Natural gas (NG) and hydrogen are the only two gases with common characteristics that remain in gaseous form at room temperature, a fact that lead their storage and transportation to difficulties (Blanco *et al*., 2016). The direct use of NG, which is mainly constitute with methane (CH_4) gas, its applications as a transportation fuel motivated by its natural clean burning characteristics in vehicular engines. It provides efficient and clean combustion, with minimal emissions of benzene, lead oxides, sulfur and solid particles compared to diesel and gasoline (Blanco *et al*., 2016, Nagarhalli *et al*., 2010). The two pronounced methods for NG storage are Liquefaction and Compression. The major stumbling block of these methods is complexity in their operation that is lowering temperature for LNG and high pressure for CNG, expensive and heavy equipment used for operation, limited refuelling stations and the high cost of refuelling with no safety guarantee to passengers on board (Beckner & Dailly, 2015). Adsorbed Natural Gas (ANG) Storage system is being considered as a promising and the best option. This is because the gas is stored on the surface of the carbonaceous material which synthesized mostly from agro-waste (precursor) (Liu *et al*., 2014). The precursors are abundant, cheap, low energy requirement, flexible pores structural modification (Biloe *et al*., 2001; Nasri *et al*., 2015), the system maintenance is cheaper and operate at relatively lower pressure and ambient temperature which reduce the risk of high pressure. Adsorbent such as zeolites, silica gels, and mesoporous solids for methane storage as been reviewed (Menon & Komarneni, 1998). Though some of these adsorbents exhibit high adsorption performance in CH_4 but they are expensive which require multi-stage fabrication (Sun *et al*., 2017). The outcome from previous researches reveal that carbon materials are more suitable for methane adsorptions due their easy tailoring texture and surface properties, low cost, availability, low energy requirement and hydrophobicity (Labus *et al*., 2014; Policicchio *et al*., 2013).

Activated carbon (AC) is basically used for purification of waste water and decontamination of air. AC is versatile in decontamination application due to its high adsorption capacity (Nasri *et al*., 2014). Applications of commercial activated carbon are limited due to its higher market prices. For the above reason, an interest has been developed in the production of AC from agro-waste. In a general perspective, conversion of non-living wastes to AC for mitigation of waste or environmental pollution is a very good idea (Saleh & Gupta, 2014). The available agro-wastes for production of AC in Malaysia includes coconut shells (Iqbaldin *et al*., 2013; Mohammed *et al*., 2015), palm kernel shells (Din *et al*., 2009; Hamza *et al*., 2016), rice husk, Kenaf, nut shells, tobacco stems and sugarcane bagasse (Din *et al*., 2009).

Coconut shell as a waste is the leading precursor used for the production of AC in Asia, which is over 60% share of the total raw materials used (Bandosz, 2006). This might be due to its availability around the region and possession of excellent properties such as high carbon

content, low in organic matter and hardness (Din *et al*., 2009; Li *et al*., 2008). Further, it constituted mainly with lignin and cellulose. In addition, coconut shells are hard, not easily powdered, which are among the good properties of precursor for activated carbon production. Poly ether ether ketone (PEEK) is aromatic, linear and semi-crystalline thermoplastic with excellent mechanical properties, which could be applied for methane storage over a wide range of pressures (Mylläri *et al*., 2015). However, the high price of PEEK limited its commercial applications such as in gas energy storage.

Most studies on PEEK focus on high temperature since it is one of the highest temperature (260ºC) resistance among all plastics (Mylläri *et al*., 2012), while studies on its mechanical properties are not common (Mylläri *et al*., 2014). Therefore, synthesis of activated carbon with high mechanical strength is desirable. Such carbon can withstand high pressure level and several adsorptions and desorption cycles without regeneration. In addition, it can minimize the cost of replacement with new adsorbent into the energy storage system. Based on the literatures reviewed, it was observed that PEEK has the desirable properties (mechanical strength). On this note, this research tailored towards investigation of hybrid PEEK and coconut shells properties on methane adsorption. Adsorption achieve only when optimum factors are employed (Hamza *et al*., 2015). This necessitates employment of RSM for optimization of the process factors for CH_4 adsorption. Experimental design is a statistical technique used for collective multi-variable optimization at a time (Ganapathy *et al*., 2011). This technique, assist in reducing the number of experimental runs, save time, costs and generate accurate and reliable results (Iqbal *et al*., 2016; Nouri-Borujerdi *et al*., 2016). Microwave heating process transfer heat around the sample through conduction and dipole rotation, the approach rises the temperature easily and distribute uniformly across the sample within a shorter period of time. To produce low-cost adsorbent with desirable properties having high mechanical properties, coconut shell was used as the main precursor blended with PEEK as core precursor with KOH as an activating agent. The objective of this research work was to apply RSM and CCD to identify the optimum conditions for preparation of stable adsorbent from coconut shell and PEEK for methane adsorption that stand higher-pressure operation and several cycles of reusability. To the best of our knowledge there is no research conducted for the preparation of adsorbent from coconut shell and PEEK using microwave for methane adsorption.

2. EXPERIMENTAL

Precursor, chemicals and other relevant materials used with procedure followed in this research work are described below:

2.1 Materials

In this study, raw coconut shell was collected from local market in Johor, Malaysia. The obtained coconut Shell (CNS) was wash severally with tap water to remove the useless particles attached to their surface. Then sun dried to remove its moisture content, followed by thermal drying using an oven at a temperature of 105ºC. Then taken out of the oven, cool to ambient temperature. After that, it was ground using a grinding machine, then sieved to a particle size of (1-2mm) and kept for carbonization. Potassium hydroxide was purchased from Sigma Aldrich. Granulated PEEK was purchased from Vitrex Tech., Lancashire UK.

2.2 SYNTHESIS OF ACTIVATED CARBON

2.2.1 Carbonization

The carbonization process was carried out using the setup shown in Figure-1. Coconut shell (CNS) was charge in a tubular reactor, then place in a lagged cylindrical vertical furnace. The material heated under a flow of nitrogen at1000mLmin^{-1} at the rate of heating of 10ºC min^{-1} from ambient temperature to 700ºC. The heating temperature held for 2 hours, then cool under a flow of nitrogen (700mLmin^{-1}) until the temperature drop below 100ºC. The furnace temperature controlled and monitored by the K-type thermocouple. After cooling, the resulting char sieved to a particle size ranges between 0.841mm-0.425mm (20- 40) mesh, then transferred into a storage bottle and kept inside desiccators to prevent moisture absorption for further use. PEEK char was also prepared by pyrolysis of granulated Victrex PEEK. All the preparation conditions are the same with CNS except for heating temperature (800ºC) and heating time (45 min).

2.2.2 Activation

The PEEK char was blended with CNS char at different percentage by weight of 15, 20, 25, 30 and 35 weight percent (wt %) of PEEK char. The KOH was mix with the blended chars in an impregnation ratio of 1.5:1 weight ratio of KOH to char. The dried mixture was activated using microwave as shown in Figure-2 at different power rate and time as suggested by design expert software but constant nitrogen flow of 200cm3/min. The activated carbon was washed to obtain neutrality state, dried in an oven, then cooled and kept in desiccators' for further use.

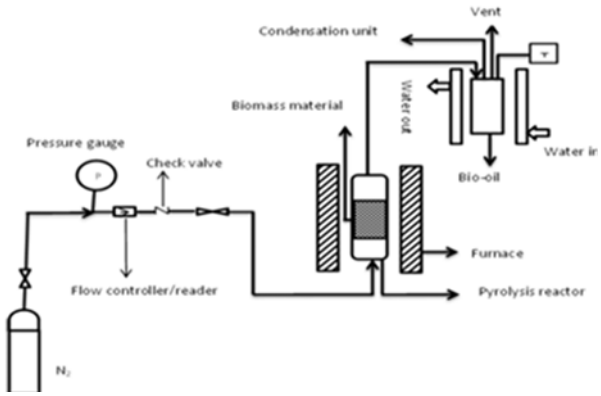

Figure-1. Schematic of carbonization rig for coconut shells and PEEK.

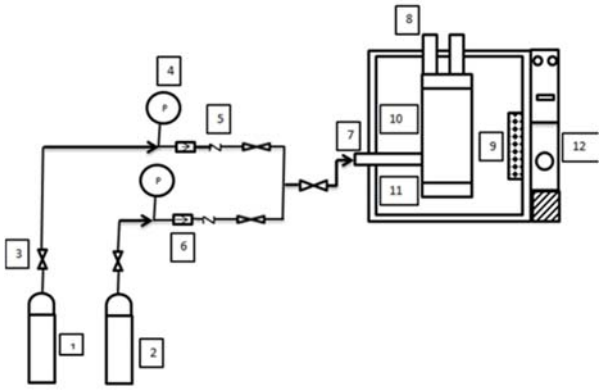

Figure-2. Microwave equipment for activation
1. CO2 gas cylinder 2. N_2 gas cylinder, 3. Control valve 4. Pressure gauge, 5. Check valve 6. Flow controller 7. Gas inlet, 8. Outlet, 9. Microwave Source 10. Microwave cavity, 11. Quartz reactor , 12. Microwave control panel

2.3 Experimental design

In this work, design expert software version 7.1.6 (Start-Ease Inc., Minneapolis, USA) used for the optimization of Sorbent preparation parameters using response surface methodology (RSM). RSM is an important tool used in experimental design for formulating a new product in which responses supported with many variables and the purpose was to optimize the responses. In this study central composite design (CCD) was used to study the preparation variables of activated carbon from coconut shells and PEEK via microwave-induced activation. Microwave power x_1 (300-700W), irradiation time x_2 (3-15min.), and quantity of PEEK char x_3, (15-35%) are the independent variables selected for this study. These variables adopted from the previous studies of(Hesas *et al*., 2013). Generally, the CCD contains 2n factorial runs, 2n axial runs then nc center runs which has shown in equation (1).

$$N = 2n+2n+nc = 20 \quad (1)$$

N is the total number of experimental runs figured out by the software which is 20, 2n is factorial runs, 2n is the axial runs and nc is the centered point, then n is the number of factors.

2.4 Methane adsorption procedure

The methane adsorption and desorption were conducted using the static volumetric unit setup as shown in Figure-3. The procedure was adopted from the work of (Fatemi *et al.,* 2011) and (Nasri *et al*., 2015). The unit was equipped with load cell, adsorption cell, K-type thermocouple, vacuum pump, pressure transducer, temperature indicators, Digital mass flow meter, and Safety valves. Prior to the adsorption measurement, the sample which was dried in-situ at 105ºC for 24 hours and cooled to room temperature was degassed in an adsorption cell at 120ºC for two hours. The load cell was fed with methane to a pressure of 7 bars while the valve between loading and adsorption cells remain closed. The initial and subsequent temperature and pressure in both cells were recorded up to the equilibrium state. The experiment commenced by opening the valve between the loading and adsorption cells for the gas to reach the adsorption cell in order to adsorbed by the adsorbent, while the valve below the adsorption cell remains closed. The equilibrium state reached when the pressure of both cells maintained constant pressure values for at least 20 minutes. The amount of gas adsorbed determined using the mass balance equation 2.

$$\frac{1}{m}\left[\left(\frac{PV}{ZRT}\right)_{L1}-\left(\frac{PV}{ZRT}\right)_{L2}+\left(\frac{PV}{ZRT}\right)_{A1}-\left(\frac{PV}{ZRT}\right)_{A2}\right]=q \quad (2)$$

Where P=Pressure, V=Volume, R=Gas constant, m=Molar weight, Z=Compressibility factor, q=Amount adsorbed, 1= the state prior to adsorption equilibrium and 2=the final equilibrium state.

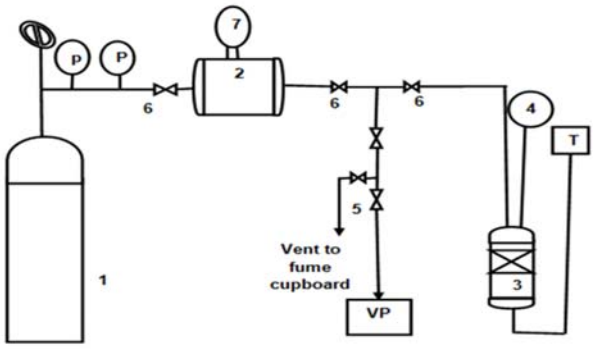

Figure-3. Schematic Diagram of the Volumetric Adsorption Set-up.
1. Natural gas cylinder, 2. Loading cell, 3. Adsorption cell 4. Digital pressure transducer, 5. Safety valves 6. Valves, 7. Analog pressure gauge, T= adsorption cell temperature, p= Gas supply pressure gauge, VP= Vacuum pump.

3. RESULTS AND DISCUSSIONS

3.1 Development of regression model equation and statistical analysis

The regression coefficient (R^2 and R^2adj.) and adequate precision values were used to identify the model fitness strength to the experimental data. The variables and response was correlated using central composite design (CCD). The response values obtained from experiment are shown in Table-1. The design expert software fits quadratic and two-factor interaction ($2F_1$). The program recommends quadratic model due to the high values of regression coefficient (R^2 and R^2adj.) and adequate precision values of 0.9511, 0.9071 and 18.701 respectively. The comparism of the average predicted error values with the predicted values ranges at the design point and signal-to-noise ratio measurement were done using precision. The adequate precision value ratio is

much more greater than 4 and is an indication of adequate model discrimination (Hesas *et al.*, 2013). The quadratic model was selected as suggested by the software for methane adsorption.

The positive and negative signs in front of the terms are indicating synergistic and antagonistic effects respectively. The model stability was justified using analysis of variance (ANOVA). The ANOVA variables for the CH_4 uptake capacity was given in Table-2. The significant of the model, standard error and the regression coefficient are all determined using the p-value and F-value. The quadratic model F-value of 21.62 and P-value of less than 0.05 indicates the quadratic model is significant. However, if the P-value is greater than 0.1 denote the model term is insignificant. The following terms for the power and time (x_1x_2), and interaction between power and amount of PEEK (x_1x_3) are all significant terms. The amount of PEEK (x_3), interaction between time and methane uptake were significant: microwave power (x_1), irradiation time (x_2), interaction between irradiation time and amount of PEEK (x_2x_3), square effect of power (x_1^2), square effect of time (x_2^2) and square effect of PEEK (x_3^2) are significant.

Table-1. Experimental design matrix and response results.

Sample Code		Actual Variable		Response	
	Run	Power x_1 (W)	Time x_2 (min)	PEEK x_3 (wt%)	Y_1 (mmol/g)
M49P25	1	400.00	9.00	25.00	5.12
M59P25	2	500.00	9.00	25.00	5.00
M59P25	3	500.00	9.00	25.00	5.00
M715P35	4	700.00	15.00	35.00	4.76
M59P25	5	500.00	9.00	25.00	5.00
M56P25	6	500.00	6.00	25.00	5.06
M315P15	7	300.00	15.00	15.00	4.92
M33P35	8	300.00	3.00	35.00	5.00
M512P25	9	500.00	12.00	25.00	4.94
M315P35	10	300.00	15.00	35.00	4.93
M69P25	11	600.00	9.00	25.00	4.98
M715P15	12	700.00	15.00	15.00	4.69
M59P20	13	500.00	9.00	20.00	4.97
M73P15	14	700.00	3.00	15.00	2.73
M59P25	15	500.00	9.00	25.00	5.00
M33P15	16	300.00	3.00	15.00	5.08
M73P35	17	700.00	3.00	35.00	4.94
M59P25	18	500.00	9.00	25.00	5.00
M59P20	19	500.00	9.00	30.00	5.06
M59P25	20	500.00	9.00	25.00	5.00

Footnote: M= microwave, P= PEEK, x_1= microwave power, x_2= irradiation time, x_3= amount of PEEK

Table-2. Analysis of variance (ANOVA) for response surface quadratic model for CH_4 uptake.

Source	Sum of Squares	df	Mean Square	F Value	p-value
Model	0.18	9	0.020	21.62	< 0.0001
x1	0.058	1	0.058	62.36	< 0.0001
x2	0.056	1	0.056	60.59	< 0.0001
x3	6.618E-004	1	6.618E-004	0.72	0.4173
x1x2	3.612E-003	1	3.612E-003	3.91	0.0763
x1x3	3.613E-003	1	3.613E-003	3.91	0.0763
x2x3	2.113E-003	1	2.113E-003	2.29	0.1616
x12	1.097E-003	1	1.097E-003	1.19	0.3016
x22	2.839E-003	1	2.839E-003	3.07	0.1103
x32	7.485E-004	1	7.485E-004	0.81	0.3894
Residual	9.245E-003	10	9.245E-004		
Lack of Fit	9.245E-003	5	1.849E-003		
Pure Error	0.000	5	0.000		
Cor Total	0.19	19			
Std. Dev.	0.030	R-Squared	0.9511		
Mean	4.97	Adj R-Squared	0.9071		
C.V. %	0.61	Pred R-Squared	0.7047		
PRESS	0.056	Adeq Precision	18.701		

3.2 Effect of the preparation variables on the methane uptake

Following the results in Table-2, the microwave power (x_1) and irradiation time (x_2) had very strong significant effect on the methane uptake with p-value<0.01. The interaction between the power and time (x_1x_2) and power with amount of PEEK (x_1x_3) had strong significant with 0.01<p-value<0.05). The amount of PEEK had less effects on the methane uptake, based on its P-value of 0.4173 which is greater than 0.05. Among the studied factors, power has the highest F-value of 62.36, followed with time with the F-value of 60.59, while amount of PEEK has the least effect with the F-value of 0.72. The interaction between power and time (x_1x_2) and the interaction between power and amount of PEEK had the same effect on the methane adsorption uptake with F-values of 3.91 and 3.91 respectively, while the interaction between times and amount of PEEK present least notable effect with F-value of 2.29. Among the quadratic functions, time had notable effect with F-value of 3.07, followed with power with F-value of 1.19, while amount of PEEK present the least F-value of 0.81.

The CH_4 adsorption performance over different variables is shown in Table-1. Determination of the optimum variables for the preparation of AC towards high methane adsorption performance was achieved using RSM. The total pressure applied for this adsorption performance is 7bar. At this pressure and at ambient temperature, the highest methane uptake achieved was 5.12 $mmolg^{-1}$. Base on the results obtain, indicate the adsorption performance of CH_4 on AC to same extent depends on the microwave power.

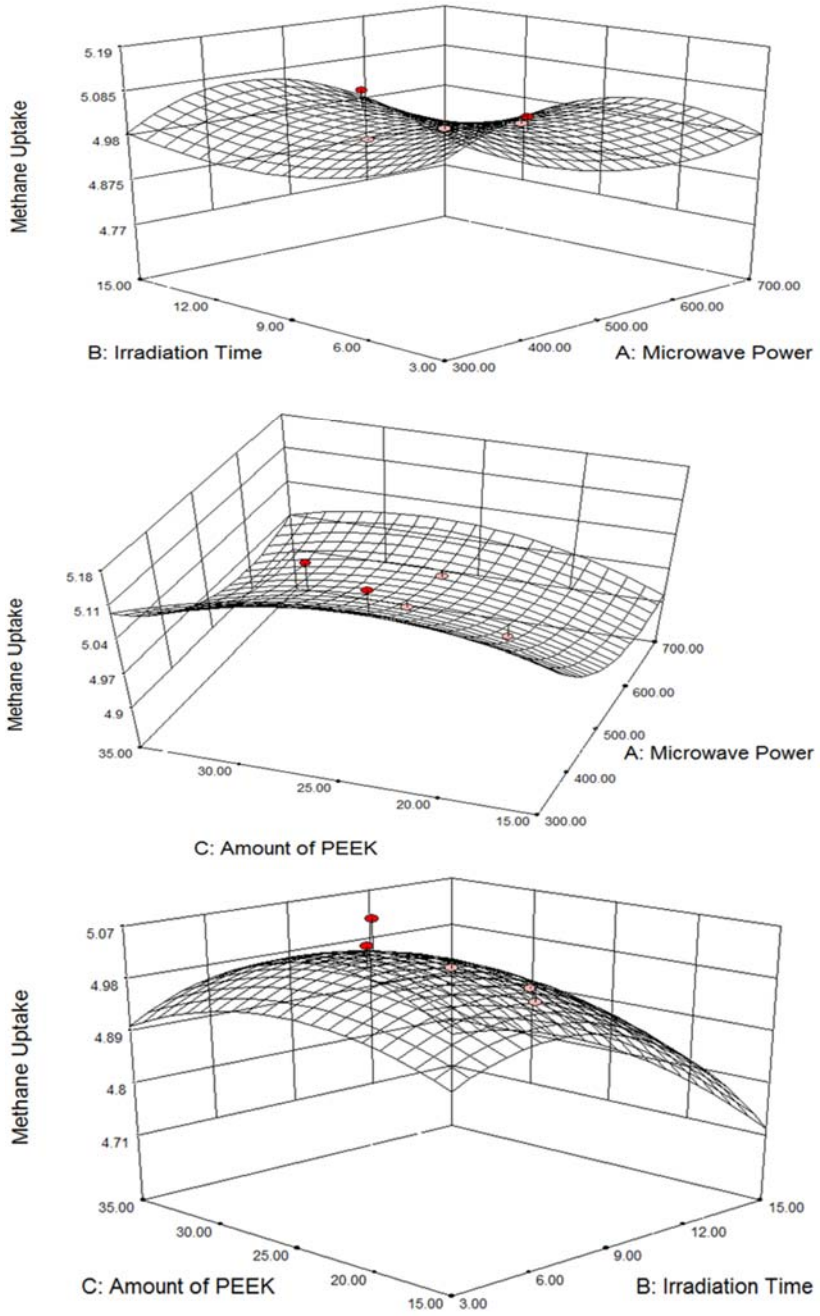

Figure-4. The three- dimensional response surface curve: (a) the effect of irradiation time and activation power, (b) the effect of amount of PEEK and irradiation time and (c) the effect of amount of PEEK and microwave power on CH4 adsorption uptake.

Figures 3 (a, b and c) shows 3D response plots which demonstrate the effect of the variables on the methane uptake. Figure-3a demonstrates the combine effect of microwave power and irradiation time, while, Figure-3b displays the effect of interaction between of irradiation time and amount of PEEK on same methane uptake, while Figure-3c displays the combine effect of amount of PEEK and microwave power on the methane uptake. According to Figure-3a the methane uptake increased appreciably with increased in activation time and power from low to moderate. Moreover, at that activation time and temperature favour the formation of pore structures as the result of the reaction of KOH with carbon species is endothermic (Liu *et al*., 2014). Further increased of both variables above the optimum decreased the uptake, it might be due to the burnt off which widen

the micropores and mesopores (Hesas *et al*., 2013). In addition, high microwave power with longer activation lead to the weight loss due to the high volatile and tar removal (Hamza *et al*., 2016). It is evidence from fig.3b that amount of has no significant effect on the methane uptake, which equally depicted in table1 of having lower F-value. The maximum methane uptake was obtained between 300 to 500W. Then further decrease as power and time of activation increase. It was reported earlier by (Hesas *et al*., 2013) that adsorption uptake increase with increase of activation power to a certain level.

4. CONCLUSIONS

Twenty experiment suggested by the software design expert version 7.1.6 and were all conducted to determine the interactive and individual effect of variable towards optimizing the response. Microwave powers, amount of PEEK and irradiation time are variables studied in this experiment. The effects of interactive and individual of all targeted variables were shown on 3-D plots. The optimum conditions for the preparation of AC of this study are: 400 microwave power, 9 minutes of irradiation time and 25wt% amount of PEEK. Power and time are played significant role than PEEK towards the CH_4 adsorption with an F-values of 62.36 and 60.59 respectively.

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