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Preparation, Optimization and Activity of Active Absorbent Synthesized from Oil Palm Ash for Flue Gas Desulfurization

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ABSTRACT

Active absorbent for the removal of SO₂ in flue gas from combustion system was prepared from oil palm ash, calcium hydroxide (Ca(OH)₂) and calcium sulfate (CaSO₄) using water hydration process. The effect of various absorbent preparation variables; hydration period, amount of oil palm ash and amount of CaSO₄ used in the preparation mixture towards the BET (Brunauer-Emmett-Teller) specific surface area of the resulting absorbent were studied using Central Composite Design (CCD). The surface area of the absorbents obtained ranges from 18.7 to 147.2 m²/g. It was found that all the three absorbent preparation variables studied exerted significant positive effect on the BET surface area of the resulting absorbent. An empirical mathematical model equation that correlates the three absorbent preparation variables to the BET surface area of the resulting absorbent was also developed. Analysis of variance (ANOVA) showed that the model was significant at a confidence level of 95%. Utilization of the model equation developed, optimum BET surface area of 128.6 m²/g was predicted at a hydration period of 10 hr, amount of oil palm ash of 15.0 g and amount of CaSO₄ of 2.7 g. The predicted optimum value agreed well with the experimentally measured values of 125.9 to 129.5 m²/g. Apart from that, activity test revealed that the absorbent derived from oil palm ash/Ca(OH)₂/CaSO₄ have a higher desulfurization capacity as compared to its starting materials.

Keywords: Absorbent; Combustion system; Flue gas; Oil palm ash; SO₂ removal.

1 INTRODUCTION

Lately, environmental regulations all over the world are becoming more restrictive concerning the control of atmospheric pollution produced in the flue gas from combustion system burning on liquid and solid fuel such as coal and oil. Control system is mainly focused on sulfur dioxide (SO₂) reduction. This is mainly due to the toxic and acidic characteristics of SO₂. Apart from being the primary cause of acid rain, which damages buildings, vegetation and water ground cycle, SO₂ also cause the formation of secondary particles in the atmosphere that impairs visibility. SO₂ is also considered to be toxic to humans by inhalation. Animal tests indicate that SO₂, although not itself carcinogenic, assists in the carcinogenic activity of other agents [1]. Therefore, there is a need to remove/reduce the SO₂ concentration in the flue gas from combustion system before releasing it to the environment using appropriate control technology.

Presently, in the practical process, SO₂ is removed by various types of flue gas desulfurization (FGD) units. A wet-type FGD unit based on a limestone-gypsum method is most widely used and suitable for large-scale boilers such as those installed in coal or oil-fired power plants. Although the wet process has a high efficiency in removing SO₂, it has a lot of disadvantages. Among some of them are the large space required for installation, the large volume of water required and the high capital and operating expenses. On the other hand, dry desulfurization process offers an attractive alternative to the wet process for FGD. Basically, dry desulfurization systems work with dry powder absorbent which reacts with the flue gas. Many studies have shown that when siliceous materials such as coal fly ash is mixed with calcium hydroxide (Ca(OH)₂) or calcium oxide (CaO) in a hydration process, absorbents with high SO₂ capture capacity could be attained [2-12]. The absorbent is formed from the pozzolanic reaction between silica (SiO₂) and/or alumina (Al₂O₃) eluted from the ash and Ca(OH)₂ or CaO to form reactive species of complex compound containing Ca, Si, Al, O ions and hydrated water which has a high surface area [2,8,12]. The structural properties of the absorbent, particularly its specific surface area, is believed to play an important role in SO₂ capture. Some studies have shown that higher desulfurization activity correlates with higher specific surface area [6,8].

This study presents the findings of using oil palm ash as the source of siliceous material for the synthesis of absorbent for flue gas desulfurization. Oil palm ash is an abundant agricultural solid waste in tropical countries like Malaysia and Thailand. The ash is produced after the combustion of oil palm fiber and shell as boiler fuel to produce steam for palm oil mill consumption. It was reported that almost 4 million tons of ash is produced annually in Malaysia. Although there are some studies on the utilization of oil palm ash such as a cement replacement material and as an absorbent for the removal of zinc from aqueous solution [13], most of

the ash are still disposed off in landfill that requires a lot of land area. Therefore, the utilization of oil palm ash becomes as essential topic to be further investigated.

In this study, a central composite design (CCD) was conducted to study simultaneously the effects of three absorbent preparation variables (hydration period, amount of oil palm ash and amount of CaSO_4) on the BET surface area of absorbent prepared from oil palm ash. From the results of the CCD, an empirical model was then developed to correlate the absorbent surface area to the absorbent preparation variables. The empirical model will be used to optimize the BET surface area of the absorbent derived from oil palm ash. The absorbent will also be tested for its activity in SO_2 absorption.

2 Experimental Section

2.1 Absorbent Preparation

The raw materials used to prepare the absorbent were oil palm ash, calcium hydroxide (Ca(OH)_2) and calcium sulfate (CaSO_4). The oil palm ash was provided by United Oil Palm Mill, Pulau Pinang, Malaysia. The chemical composition of oil palm ash was determined using Rigaku RIX 3000 X-ray Fluorescence (XRF) spectrometer and is given in Table 1. The Ca(OH)_2 and CaSO_4 were obtained from BDH Laboratories, England. The BET specific surface area (measured on an Autosorb 1C Quantachrome analyzer) of these raw materials is given in Table 2.

Table 1 : Chemical Composition of Oil Palm Ash

Composition	Percentage (%)
SiO_2	40.0
Al_2O_3	6.1
CaO	10.0
Fe_2O_3	2.5
MgO	6.4
K_2O	12.1
P_2O_5	8.2
C	5.4
Others	2.0
Ignition loss	7.3

Table 2 : BET Specific Surface Area of Starting Materials

Starting material	Specific surface area (m^2/g)
Oil palm ash	10.2
Ca(OH)_2	6.2
CaSO_4	4.9

For the preparation of the absorbent, 5 g of Ca(OH)_2 were added to 100 ml of water at 65 °C. Upon stirring, the temperature of the slurry increased to about 80 °C. Specific amount of oil palm ash and CaSO_4 were added to the slurry simultaneously. The slurry was then heated at 100°C for a period of time in order for the hydration process to occur. The resulting slurry was filtered and dried at 200 °C for 2 hr. The absorbent in powder form were then palletized, crushed and sieved to produce the required particle size range (250-300 μm). The BET surface area of the absorbents were analyzed as described above. The hydration period, amount of oil palm ash and amount of CaSO_4 were varied according to an experimental design described below.

2.2 Experimental Design

The experimental design chosen for this study was a Central Composite Design (CCD) that helps in investigating linear, quadratic and cross-product effects of the three absorbent preparation variables on the specific surface area of the absorbent [14]. The three absorbent preparation variables studied are hydration period, amount of oil palm ash and amount of CaSO_4 . Table 3 list the range and levels of the three absorbent preparation variables studied. The CCD comprises a two-level full factorial design ($2^3 = 8$), six axial or star points and six center points. The value of α for this CCD is fixed at 1.68 [14]. The complete design matrix of the experiments employed and results are given in Table 4. All variables at zero level constitute to the center

points and the combination of each of the variables at either its lowest (-1.68) level or highest (+1.68) level with the other variables at zero level constitute the axial points. The experiment sequence was randomized in order to minimize the effects of the uncontrolled factors.

Table 3 : Levels of the Absorbent Preparation Variables chosen for this Study

Variable	Coding	Units	Levels				
			-1.68	-1	0	+1	+1.68
Hydration period	x_1	hr	3.18	10.0	20.0	30.0	36.82
Amount of oil palm ash	x_2	g	1.6	5.0	10.0	15.0	18.4
Amount of CaSO ₄	x_3	g	0.32	1.0	2.0	3.0	3.68

Table 4 : Experimental Design Matrix and Results

Solid code	Type	Experimental variables			BET surface area (m ² /g)
		Hydration period, x_1 (hr)	Amount of oil palm ash, x_2 (g)	Amount of CaSO ₄ , x_3 (g)	
A1	Center	0	0	0	55.2
A2	Center	0	0	0	54.4
A3	Center	0	0	0	50.3
A4	Center	0	0	0	55.1
A5	Center	0	0	0	56.4
A6	Center	0	0	0	52.7
A7	Axial	0	-1.68	0	26.0
A8	Axial	0	0	-1.68	41.3
A9	Axial	-1.68	0	0	18.7
A10	Axial	1.68	0	0	134.2
A11	Axial	0	1.68	0	105.9
A12	Axial	0	0	1.68	142.4
A13	Factorial	1	-1	1	68.8
A14	Factorial	1	1	-1	126.0
A15	Factorial	-1	1	-1	55.6
A16	Factorial	1	1	1	147.2
A17	Factorial	-1	1	1	128.6
A18	Factorial	-1	-1	1	35.8
A19	Factorial	-1	-1	-1	50.2
A20	Factorial	1	-1	-1	71.7

Each response of the specific surface area in Table 4 was used to develop an empirical model that correlates the specific surface area to the absorbent preparation variables using a second-degree polynomial equation as shown in Equation (1),

$$Y = b_0 + \sum_{i=1}^3 b_i x_i + \left(\sum_{i=1}^3 b_{ii} x_i \right)^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij} x_i x_j \quad (1)$$

where Y is the predicted specific surface area (m²/g), b₀ is the constant coefficient, b_i are the linear coefficients, b_{ij} are the interaction coefficients, b_{ii} are the quadratic coefficients and x_i, x_j are the coded values of the absorbent preparation variables.

2.3 Model Fitting and Statistical Analysis

Design Expert software version 6.0.6 (STAT-EASE Inc., Minneapolis, USA) was used for regression analysis of the experimental data to fit the second-degree polynomial equation and also for evaluation of the statistical significance of the equation developed. The software was also used to identify the optimum surface area within the range of absorbent preparation variables studied.

2.4 Desulfurization Activity Study

Desulfurization experiments were performed using a fixed bed reactor. The reaction zone was contained in a 0.01 m inner diameter stainless steel tube fitted in a furnace for isothermal operation. The absorbent (0.7 g)

was packed in the center of the reactor supported by glass wool. A stream of feed gaseous mixture containing 2000 ppm of SO₂, 500 ppm of NO, 12% of CO₂, 5% of O₂ and balance nitrogen at a reaction temperature of 100°C was passed through the absorbent. Prior to that, the N₂ gas stream was humidified using a humidification system, which consists of two 250 ml conical flask immersed in a water bath at constant temperature. The total flow rate of the gas stream was controlled at 150 ml/min using mass flow controller. The details of the experimental set-up is reported elsewhere [15]. The concentration of the SO₂ in the flue gas was measured using a Portable Flue Gas Analyzer IMR2800P before and after the absorption process. The concentration of SO₂ was recorded continuously until 60 min. Two or three replicate measurements were made for each activity test. For clarity, only the averages are presented in this paper. The activity of the absorbent is reported in terms of percent SO₂ removal as shown in Equation (2).

$$\text{Percent removal of SO}_2 (\%) = \frac{\{[\text{SO}_2]_{\text{inlet}} - [\text{SO}_2]_{\text{outlet}}\}}{[\text{SO}_2]_{\text{inlet}}} \times 100\% \quad (2)$$

3 RESULTS AND DISCUSSION

3.1 Development of Empirical Mathematical Model Equation

A central composite design (CCD) was used to develop a correlation between the absorbent preparation variables to the surface area of the absorbent. The complete design matrix and surface area responses at various absorbent preparation variables are listed in Table 4. As can be seen from Table 4, the surface area obtained range from 18.7 to 147.2 m²/g. Run A1 to A6 at the center point of the design were used to determine the experimental error. As the result of surface area of these six runs were quite consistence, single replicate experimental run is essential in this study. By using multiple regression analysis, the response (surface area) obtained in Table 4 was correlated with the three absorbent preparation variables studied using the polynomial equation as shown in Equation (1). The coefficients of the full empirical model equation and their statistical significance were determined and evaluated using Design-Expert 6.0.6 software. The final model in terms of coded value after excluding the insignificant terms (identified using Fisher's Test) is as shown in Equation (3),

$$Y = 54.3 + 24.7x_1 + 26.7x_2 + 18.1x_3 + 9.0x_1^2 + 5.3x_2^2 + 14.5x_3^2 + 14.0x_2x_3 \quad (3)$$

The quality of the model developed could be evaluated from the coefficients of correlation. The value of R² for Equation (3) is 0.8929. It implies that 89.3% of the total variation in the surface area responses is attributed to the experimental variables studied.

3.2 Model Adequacy Check

The adequacy of the model was further checked with analysis of variance (ANOVA) as shown in Table 5. Based on a 95% confidence level, the model was tested to be significant as the computed F value (14.29) is much higher than the theoretical F_{0.05(7,12)} value (2.91), indicating that the empirical model is reliable in predicting the absorbent surface area. From these statistical tests, it was found that the model is adequate for predicting the absorbent specific surface area within the range of the absorbent preparation variables studied.

3.3 Effects of Absorbent Preparation Variables

The results in Table 4 show that the absorbent preparation variables have great effect on the absorbent surface area. It was found that the hydration process always improve the surface area of the resulting absorbent as compared to the raw materials. These results illustrate that there is a great possibility in improving the surface area of the absorbent with proper selection of absorbent preparation variables. As mentioned earlier, absorbent surface area may be the key factor to obtain absorbent with high desulfurization activity.

From the empirical model shown in Equation (3), it was observed that all the three individual absorbent preparation variables exerted significant influence on the absorbent surface area. Since the magnitude of the coefficient of the three individual variables are quite close, thus it can be concluded that the effect of the three variables on the surface area is equally the same. Apart from that, the quadratic term of the three absorbent preparation variables also effect the absorbent surface area significantly, but less pronounced than its linear term. Besides that, the effect of interaction between variables x₂ and x₃ also effect the absorbent surface area significantly.

Table 5 : Analysis of Variance (ANOVA) for the Regression Model Equation and Coefficients

Source	Sum of squares	Degrees of freedom	Mean of square	F-test
Model	28131.92	7	4018.85	14.29
x_1	8348.49	1	8348.49	29.68
x_2	9765.78	1	9765.78	34.72
x_3	4463.77	1	4463.77	15.87
x_1^2	1172.84	1	1172.84	4.17
x_2^2	405.37	1	405.37	1.44
x_3^2	3011.35	1	3011.35	10.71
x_2x_3	1556.26	1	1556.26	5.53
Residual	3375.56	12	281.30	-
Total	31507.48	19	-	-

Fig. 1 shows the effect of hydration period and amount of oil palm ash on the absorbent surface area. The amount of CaSO_4 used was held fix at zero level (2g). It was found that higher hydration period and amount of oil palm ash resulted in absorbents with higher surface area. In the reactions involving siliceous materials such as oil palm ash, it was reported that pozzolanic reactions typically starts with the digestion of vitreous silica and/or alumina by alkaline water and this process was reported to be very slow [8]. The dissolves silica and/or alumina will then reacts with $\text{Ca}(\text{OH})_2$ to form the reactive species in the absorbent that absorb SO_2 . Therefore, it can be concluded that in order to produce absorbent from oil palm ash that has a high surface area, sufficient hydration period of time is required. This is because, at a longer hydration period, more silica and/or alumina will dilutes out from oil palm ash to react with $\text{Ca}(\text{OH})_2$ to form the reactive species. On the other hand, the amount of oil palm ash generally determines the amount of silica and alumina available in the preparation mixture for reactions with $\text{Ca}(\text{OH})_2$ to form the reactive species in the absorbent. Thus, at a higher amount of oil palm ash used, there will be more silica and alumina available in the preparation mixture to react with $\text{Ca}(\text{OH})_2$ completely to form the reactive species. Thus, this will results in a positive effect on the surface area of the absorbent.

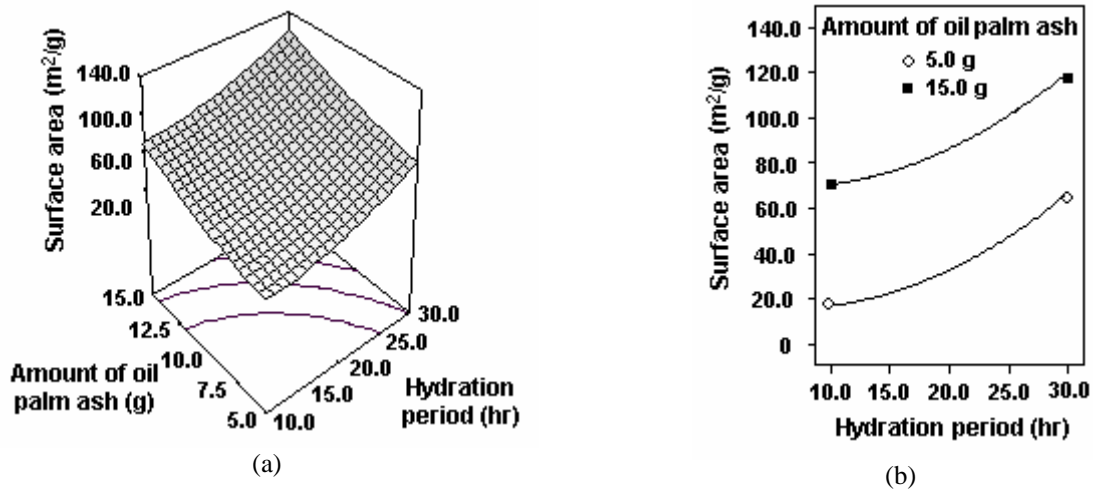


Figure 1 : Effect of Amount of Oil Palm Ash and Hydration Period on the Absorbent Surface Area; (a) Response Surface Plot and (b) Two Dimensional Drawing

Fig. 2 shows the changes in the absorbent surface area with varying amount of CaSO_4 and amount of oil palm ash. The hydration period was held fix at zero level (20 hr.). The amount of CaSO_4 used in the preparation mixture was found to give a positive effect on the absorbent surface area. The positive effect of CaSO_4 on the surface area of absorbent prepared from other siliceous material such as coal fly ash has been reported by previous researcher [16]. It was reported that this phenomena is due to the role played by CaSO_4 where it promotes the reactivity of $\text{Ca}(\text{OH})_2$ towards silica and/or alumina by suppressing crystal growth of $\text{Ca}(\text{OH})_2$. Based on the results obtained in this study, thus it could be concluded that CaSO_4 has the same effect on absorbent prepared from oil palm ash as on absorbent prepared from coal fly ash. From the empirical model shown in Equation (3), it was found that the effect of interaction between variables x_2 (amount of oil palm ash) and x_3 (amount of CaSO_4) also effect the absorbent surface area significantly. The interaction effect of

these two variables are shown clearly in Fig. 2b. The effect of the amount of oil palm ash on the absorbent surface area is more significant when more CaSO_4 was used in the preparation mixture. The following explanation describes this phenomena. When lesser amount of CaSO_4 is used in the preparation mixture, there is limited amount of CaSO_4 that could keep the reactivity of Ca(OH)_2 towards silica and/or alumina. Thus, increasing the amount of oil palm ash used in the preparation mixture do not have significant effect on the absorbent surface area as the limited amount of reactive Ca(OH)_2 becomes the limiting factor for the formation of reactive species in the absorbent (the formation of more reactive species will increase the surface area of the absorbent). However, at a higher amount of CaSO_4 used, the amount of reactive Ca(OH)_2 is no longer the limitation factor for the formation of the reactive species in the absorbent. Thus, increasing the amount of oil palm ash will increase the formation of the reactive species (more silica and/or alumina can react with Ca(OH)_2) and thus resulted in a more significant increase in the absorbent surface area.

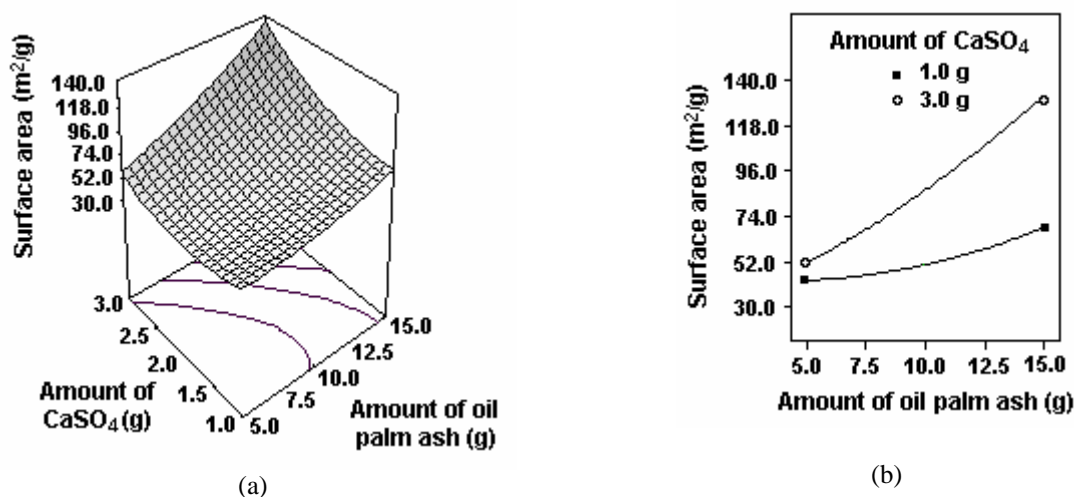


Figure 2 : Effect of Amount of CaSO_4 and Amount of Oil Palm Ash on the Absorbent Surface Area; (a) Response Surface Plot and (b) Two Dimensional Drawing

3.4 Optimization

Based on the data presented in Table 4, it was found that a maximum specific surface area of $147.2 \text{ m}^2/\text{g}$ exists within the range of the hydration process variables investigated. The hydration conditions that result in the maximum surface area are hydration period (x_1) = 30 hr., amount of oil palm ash (x_2) = 15.0 g and amount of CaSO_4 (x_3) = 3.0 g. Although the absorbent with the predicted maximum surface area would have a very high desulfurization capacity, a major drawback of the identified hydration conditions is that a hydration period of 30 h is required. Such a long period of hydration is unlikely to be used in full-scale production of the absorbents due to practical constraints and unfavourable economic considerations. Thus, it was decided to fix the hydration period (x_1) at 10 hr and optimize the other two hydration process variables (x_2 and x_3). Using the point prediction function given in Design Expert v6.0.6, it was predicted that a maximum surface area of $128.6 \text{ m}^2/\text{g}$ can be obtained using 15.0 g of oil palm ash and 2.7 g of CaSO_4 . In order to verify the prediction, two experiments were carried out under the condition predicted and absorbents with surface area of 125.9 and $129.5 \text{ m}^2/\text{g}$ were obtained. These results are in excellent agreement with the predicted value, and therefore validate the effectiveness of the empirical model in predicting/optimizing the absorbent surface area within the range of variables studied.

3.5 Desulfurization Activity

The extents of SO_2 removal by the absorbent prepared using oil palm ash and the three starting materials are shown in Fig. 3. The absorbent with the optimized surface area is used in this activity test. The fixed bed reactor was subjected to a feed gas consisting of 2000 ppm SO_2 , 500 ppm NO , 5% O_2 , 12% CO_2 and balance N_2 at 100°C . As can be seen in Fig. 3, the absorbent prepared from oil palm ash easily outperformed its base components in removing SO_2 . The absorbent removed 100% of the SO_2 in the feed gas during the first 12 min of reaction. The SO_2 started to breakthrough at reaction time > 12 min, resulting in a gradual increase in the outlet concentration of SO_2 from the reactor. In contrast, each of the three starting materials; oil palm ash, Ca(OH)_2 , and CaSO_4 , did not exhibit any significant desulfurization activity. These results mostly likely reflect the fact that the specific surface area of the absorbent prepared from oil palm ash ($128.6 \text{ m}^2/\text{g}$) was much larger than those of the starting materials (4.9 to $10.2 \text{ m}^2/\text{g}$).

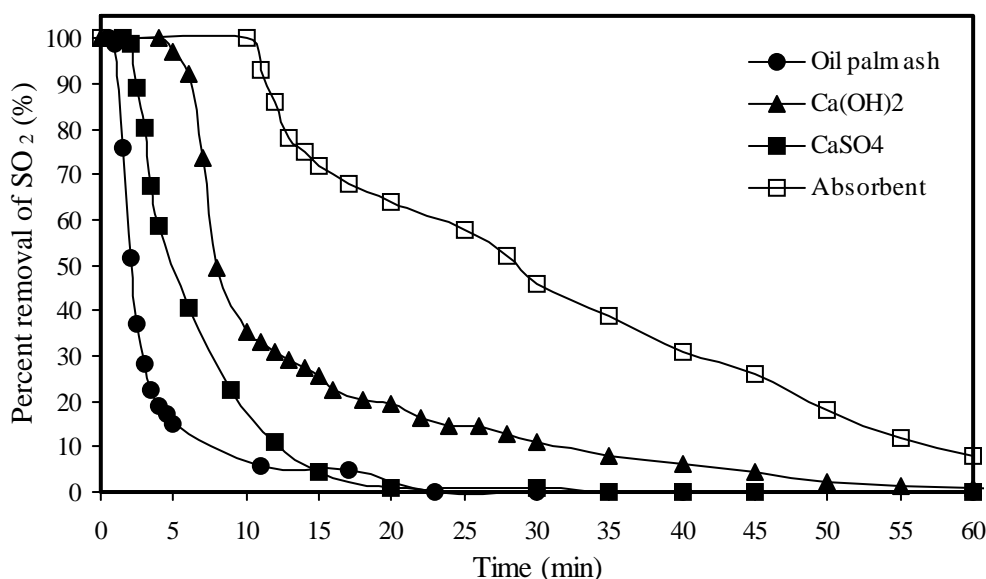


Figure 3 : Comparison of the Desulfurization Activity of the Absorbent And its Base Components. Feed Gas Composition at 100 °C: 2000 ppm SO₂, 500 ppm NO, 5% O₂, 12% CO₂, and the balance N₂.

4 CONCLUSION

Active absorbents for the removal of SO₂ in flue gas from combustion system were prepared from oil palm ash, Ca(OH)₂ and CaSO₄ using water hydration. A central composite design (CCD) was used to develop an empirical model that correlates the absorbent surface area with the absorbent preparation variables. Higher hydration period (x_1), amount of oil palm ash (x_2) and amount of CaSO₄ (x_3) used in the preparation mixture was found to give a positive effect on the absorbent surface area. The empirical model was then used to predict the optimum conditions for the preparation of absorbent with the highest surface area. The predicted value was found to agree very well with the experimental results. The desulfurization activity of the absorbent derived from oil palm ash/Ca(OH)₂/CaSO₄ was found to be significantly higher than its starting materials.

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