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Interaction of Taguchi Design with Quantitative Analysis of Crystalline Phase in Triaxial Ceramic Employing Palm Oil Fuel Ash Application

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Abstract: Application of triaxial ceramic commonly performed by crystalline phase analysis using X-ray diffraction (XRD) analysis. In this paper, Taguchi L₁₈ orthogonal array layout was used as an experimental design to perform quantitative analysis of crystalline phase test. Palm Oil Fuel Ash (POFA) was employed as a secondary filler material in the triaxial ceramic composition. The parameters investigated were the type of POFA, POFA composition, molding pressure, sintering temperature, and soaking time. Meanwhile, the responses analyzed include the percentage of mullite and albite phases, where it was objected by criterion higher is better and smaller is better, respectively. The Taguchi design analysis had suggested that the highest mullite and smallest albite phase specimens were obtained at 5 wt. % of unground POFA (GPOFA), 3-ton (4-ton for albite phase) of molding pressure, 1200 °C of sintering temperature, and 180 min of soaking time.

1. Introduction

POFA is daily wastage material from the boiler of palm oil industry. POFA is collected merely due to the second largest palm oil productions achieved by Malaysia after Indonesia [1]. The unmanageable of POFA in the landfills has caused environmental problems. The researchers have explored POFA in various benefit applications, and mostly it has applied in the concrete applications [2–6]. Previously, POFA has used as a filler and pozzolanic material in cement. On another hand, POFA has also used in another application as a barrier to an electromagnet interference [7,8]. POFA has studied in various conditions for a particular application, and the different performance properties have obtained. Well known, original POFA has an irregular shape with inconsistent particle sizes. Therefore, researchers had sieved and milled the original POFA to form UGPOFA and ground POFA (GPOFA). Triaxial ceramic typically prepared by plastic material, flux agent, and filler material. Presently, researchers explore several types of waste materials as secondary raw material such as coal fly ash [9], rice straw ash [10], EAF stainless steel slag [11], high alumina fly ash [12], others. The influences of those materials to the respective responses profoundly explored especially for the physical and mechanical properties such as density, porosity, and strength. Many types of studies have reported by
previous researchers to develop new optimal parameters. The familiar methods are the design of experiments such as Taguchi design, mixture design, and response surface methodology. Presently, the study of triaxial ceramic has applied Taguchi design to develop optimal parameter for peak temperature, dwell time, and heating rate for high temperature Co-fired ceramics application regarding responses of surface roughness, shrinkage, and density results [13]. Particularly, mixture design has studied to develop an optimal raw material composition, where ternary diagram typically used [14–16]. Response surface methodology has studied similarly with Taguchi design which is developing optimal parameters from several types of parameters, but it typically related to the 3-D plots [17]. Taguchi design has known as an effective design of experiment for developing new optimal parameters. The smaller experimental number has presented by Taguchi design compared to others. Recently, the study of the porous membrane synthesis has applied nine experiments instead of 27 experiments from factorial design [18]. The higher differences of experimental numbers have presented by a study of the synthesis and characterization of geopolymers produced using treated POFA (TPOFA), where it is applying 25 runs instead of 15625 runs if factorial design considered [19]. It is indeed consuming higher cost and time to run those factorial design with the proper experimental process. However, all these Taguchi design studies have applied if the responses categorized as numerical. The criterion of the signal to noise (SN) ratio in Taguchi design analysis needs the numerical value to classify each response regarding criterion.

Mostly, XRD analysis provides the plots for the researchers to estimate the phases provided by a specimen. Well known, Taguchi design needs numerical results for the further analysis process. Recently, XRD results have studied several types of design of experiment with quantitative results. Hence, the present study explores the interactions developed between Taguchi design with quantitative analysis of crystalline phase in triaxial ceramic employing POFA application.

2. Methodology

2.1. Design of experiment using Taguchi design

The experimental work used strictly follows the conventional industrial practice. However, given the available laboratory facilities, such procedures did not include. Furthermore, present work was conducted according to Taguchi orthogonal array layout. The raw materials used were kaolin, feldspar, and quartz as primary raw materials for the triaxial ceramic specimens. Kaolin (grade KM40) was supplied by Kaolin (M) Sdn Bhd, Malaysia. Besides, feldspar and quartz were provided by Sibelco Sdn Bhd, Malaysia. Meanwhile, original POFA was collected from Genting Ayer Hitam Oil Mill, Johor, Malaysia. In triaxial ceramic composition, applied POFA was collected from POFA layer formation process [22]. Therefore; kaolin, feldspar, quartz, and that selected POFA were used as raw materials in this triaxial ceramic application. The chemical composition of these raw materials is shown in Table 2.

2.2. Experimental works

The list of authors should be indented 25 mm to match the abstract. The style for the names is initials then surname, with a comma after all but the last two names, which are separated by ‘and’. Initials should not have full stops—for example A J Smith and not A. J. Smith. First names in full may be used if desired. If an author has additional information to appear as a footnote, such as a permanent address or to indicate that they are the corresponding author, the footnote should be entered after the surname.

| Table 1. Experimental design from Taguchi L18 orthogonal array | Run | Parameters |
|---------------------------------------------------------------|-----|------------|
|                                                              | Type of POFA | POFA composition | Molding pressure | Sintering temperature | Soaking time |
|                                                              |               | (wt. %)          | (ton)            | (ºC)                  | (min)       |
| 1                                                              | UGPOFA        | 5                | 2                | 800                   | 60          |
| 2                                                              | UGPOFA        | 5                | 3                | 1000                  | 180         |
Table 2. Chemical composition of raw materials

| Raw Material | oxide compositions (wt. %) | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | Na₂O | K₂O |
|--------------|----------------------------|------|-------|-------|-----|-----|------|------|
| Kaolin       |                            | 53.99| 41.48 | 1.49  | -   | 0.49| -    | 2.55 |
| Feldspar     |                            | 69.51| 17.71 | 0.39  | 2.73| -   | 5.38 | 4.27 |
| Quartz       |                            | 98.40| 1.12  | -     | -   | -   | -    | 0.48 |
| POFA         |                            | 64.75| 7.71  | 6.88  | 8.65| 3.11| -    | 8.90 |

The first parameter, the type of POFA was performed by dividing selected POFA layers into UGPOFA and GPOFA. Selected POFA layers particles were sieved in 160 µm siever to form UGPOFA. It was then ground in a ball mill (250 rpm and 30 min) and re-sieved in 63 µm siever to form GPOFA. Accordingly, mixing process was performed to fulfill second parameter (POFA composition). The ternary system of 50:25:25 was used for kaolin-feldspar-quartz composition. Two types of POFA and three POFA compositions provided six different compositions for mixing process as shown in Table 3. The mixing process was performed at 250 rpm and 30 min at a constant 100 g of amounts.

Table 3. Percentage composition of raw materials

| No. | Percentage Composition of Raw Materials (wt. %) | Kaolin | Feldspar | Quartz | UGPOFA | GPOFA |
|-----|-----------------------------------------------|--------|----------|--------|--------|-------|
| 1   |                                               | 50     | 25       | 20     | 5      | -     |
| 2   |                                               | 50     | 25       | 10     | 15     | -     |
| 3   |                                               | 50     | 25       | -      | 25     | -     |
| 4   |                                               | 50     | 25       | 20     | -      | 5     |
| 5   |                                               | 50     | 25       | 10     | -      | 15    |
| 6   |                                               | 50     | 25       | -      | -      | 25    |

The molding pressure as the third parameter was performed by forming process. The molding pressure is the pressure level that applied to the powders. The specimens used in this work were cylindrical pellet (diameter~13mm, thickness~3-5mm). The specimens were then sintered in an electric furnace to fulfill the fourth and fifth parameters. The sintering temperature is the highest temperature and the soaking time is the time the highest temperature held. Three different sintering temperatures and three different soaking times provided nine conditions of the sintering process, where the heating and cooling rates were set constant (5 °C/min).

The specimens were then performed using XRD (Bruker D8 Advance). It was operated at 40 kV and 40 A. The specimen was placed at the center of the diffraction chamber. The specimen was then exposed to the Cu Kα radiation (λ=1.5404 Å) in reflection mode and reflections were recorded by the
detector. When the sample rotates in the same direction at half angular velocity \( \frac{\theta}{2} \) min-1, the sample rotates in the same direction at a half angular velocity of the detector, \( \theta \), to keep the detector at the focusing point of the diffracted X-rays. The chart recorder recorded the intensities and \( \frac{\theta}{2} \) values of the diffracted peaks simultaneously with that rotation. The peak identifications and quantitative analysis of the different crystalline phases were finally estimated. In this research, DIFFRAC.EVA software and PDF-4 ICDD database were used. In the analysis of crystalline phase, similar crystalline phase database was used to distinguish appropriate quantitative evaluations. The results were briefly analyzed using MINITAB 17 software. The mullite and albite phases percentages were classified as output responses in that software.

3. Results and discussion

3.1. Design of experiment using Taguchi design

The XRD plots were analyzed based on similar crystalline phases. The crystalline phases involved are Quartz (ICDD:00-046-1045), Mullite (ICDD:01-074-4146), Albite (ICDD:00-009-0466), and Cristobalite (ICDD:01-071-6240). Table 4 shows the result of quantitative analysis of crystalline phases and its mullite phase percentage rank. It is evaluated based on these similar four crystalline phases to control the precision of mullite ranking among Taguchi L18 orthogonal array specimens.

| No. | Crystalline phase | Quartz (00-046-1045) | Mullite (01-074-4146) | Albite (00-009-0466) | Cristobalite (01-071-6240) | Rank (1: Highest) |
|-----|------------------|----------------------|----------------------|---------------------|-----------------------------|-------------------|
| 1   |                  | 62.5                 | 0.0                  | 37.5                | 0.0                         | 13*               |
| 2   |                  | 51.8                 | 25.3                 | 20.5                | 2.4                         | 8                 |
| 3   |                  | 49.4                 | 38.7                 | 5.0                 | 6.9                         | 4                 |
| 4   |                  | 55.1                 | 0.0                  | 44.9                | 0.0                         | 13*               |
| 5   |                  | 49.1                 | 12.6                 | 35.3                | 5.2                         | 9                 |
| 6   |                  | 49.3                 | 38.0                 | 7.2                  | 5.6                         | 6                 |
| 7   |                  | 44.8                 | 10.6                 | 41.6                | 3.0                         | 10                |
| 8   |                  | 37.9                 | 43.8                 | 10.4                | 7.9                         | 2                 |
| 9   |                  | 50.7                 | 0.0                  | 49.3                | 0.0                         | 13*               |
| 10  |                  | 49.5                 | 38.3                 | 6.3                  | 6.0                         | 5                 |
| 11  |                  | 63.7                 | 0.0                  | 36.3                | 0.0                         | 13*               |
| 12  |                  | 51.3                 | 32.5                 | 13.3                | 3.0                         | 7                 |
| 13  |                  | 54.6                 | 10.5                 | 32.3                | 2.6                         | 11                |
| 14  |                  | 47.9                 | 38.7                 | 8.9                  | 4.5                         | 3                 |
| 15  |                  | 58.6                 | 0.0                  | 41.4                | 0.0                         | 13*               |
| 16  |                  | 36.6                 | 44.4                 | 12.6                | 6.4                         | 1                 |
| 17  |                  | 50.1                 | 0.0                  | 49.5                | 0.4                         | 13*               |
| 18  |                  | 49.3                 | 4.4                  | 43.7                | 2.6                         | 12                |

Note: * Refer to similar mullite ranking because all these specimens show zero mullite percentage

Mullite phase is entirely focused on evaluating the performance properties of sintered specimens. In Taguchi L18 orthogonal array, the highest mullite phase percentage is presented by the No.16 specimen (44.4 %), where it is prepared by 25 wt. % of GPOFA, 2-ton of molding pressure, 1200 °C of sintering temperature, and 180 min of soaking time. In the meantime, five further specimens that showed a high mullite percentage were also given by 1200 °C temperature. However, these six specimens have a different level of other parameters. It concludes in the range of 800-1200 °C; the mullite phase percentage increased with temperature increased. The results of quantitative analysis also conclude that highest and lowest mullite phase specimen of 1200 °C sintering temperature is the No.16 and No.6 specimens, respectively. In the meantime, the highest and lowest mullite phase specimen of 1000 °C is the No.12 and No.18 specimens, respectively. Nevertheless, all specimen of 800 °C show zero mullite phase percentage. Thus, it is
evaluated based on the albite phase percentage. The results show that lowest and highest albite phase are presented by the No.11 and No.17 specimens, respectively. Therefore, these six specimens have primarily compared each other regarding its XRD plots in Figure 1. It is sorted according to sintering temperature values. In each temperature, two specimens are selected to have compared each other. The mullite phase peak continually observed decreased when the temperature dropped while the albite phase peak reduced when the temperature increased. Well presented, the mullite phase is not shown on all 800 °C specimens. Thus, Figure 1(e) and Figure 1(f) have not demonstrated by any mullite peaks. In contrary, these two plots are highly presented by the higher albite phase peaks. The mullite phase peaks are only observed in 1000 °C and 1200 °C specimens as shown in Figure 1(a)-(d). The No.18 specimen is the lowest mullite percentage among 1000 °C specimens. This specimen is still surrounded by the massive albite phase peak as shown in Figure 1(d). Meanwhile, the highest mullite percentage specimen among 1000 °C specimens are assigned by the No.12 specimen.

Figure 1. XRD plots of Taguchi L18 orthogonal array specimens

Figure 2 presents the interaction of mullite and albite percentages according to Taguchi L18 orthogonal array specimens. Specifically, the label for X-axis is sorted according to mullite phase percentage for first twelve specimens, while albite phase for six remaining specimens. Generally, trends of line graph for mullite and albite phases have reported opposed each other. In terms of mullite phase, it is increased with increasing of temperature. Particularly, Conconi et al., (2014) has shown that mullite peaks initially appear after the higher temperatures [23]. Meanwhile, albite is mostly decreased with increasing of temperature. However, two specimens of 1000 °C have higher albite phase percentage than specimens of 800 °C, which is No.7 and No.18 specimens. The No.7 specimen is prepared by 25 wt.% of UGPOFA, pressed at 2-ton, and 60 min of soaking time. Meanwhile, the No.18 specimen is prepared by 15 wt.% of GPOFA, pressed at 4-ton, and 60 min of soaking time. Another four specimens of 1000 °C, are typically soaked by 180 and 300 min. Higher albite phase for these two specimens (No.7 and No.18 specimens) is generally caused by 60 min of soaking time. This 60 min of soaking time typically produces minimum energy in the sintering process, which lowering rate of disappearing process of albite phase. Hence, there is much more albite remained for 60 min of soaking time compare to the longer soaking time specimens of 1000 °C.
Particularly, these two specimens (No.7 and No.18 specimens) have higher albite content than three specimens of 800 °C sintering temperature, which are No.11, No.1, and No.15 specimens. These three specimens apparently have the lower energy for mullite formations compared to No.7 and No.18 specimens. But the rate of disappearing process of albite phase in a specimen might be higher than No.7 and No.18 specimens. In the meantime, both No.11 and No.1 specimens are sintered at the similar soaking time which is 60 min. Hence, the condition of minimum sintering temperature and soaking time are also shown by these two specimens. Moreover, these two specimens are the fastest process in this triaxial ceramic process. In the sintering process, smallest power is used by these two specimens. In this condition, the specimens could be having lowest mullite phase content and highest albite phase content. In terms of albite phase, the results obtained are opposite, where those two specimens have smallest albite phase although soaked at the minimum soaking time. Furthermore, No.15 and No.4 specimens are apparently reported soaked by 180 min No.9 and No.17, while No.9 and No.17 specimens are soaked by 300 min. Specific to 800 °C specimens, the albite phase is decreased with decreasing of soaking time.

3.2. Taguchi design analysis for optimal parameters development

Mainly, Taguchi design deals with SN ratio analysis. In this study, mullite and albite phases percentages were classified as ‘Larger is better’ and ‘Smaller is better’ criterion, respectively. However, for mullite phase percentage results, all zero-value was assumed as one to prevent error during SN ratio calculation because it deals with ‘Larger is better’ criterion. The SN ratio for mullite and albite phases percentages are calculated by Eq. (1) and (2), respectively.

\[
\text{SN ratio}_{\text{Mullite}} = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2} \right)
\]  

(1)

\[
\text{SN ratio}_{\text{Albite}} = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} y_i^2 \right)
\]  

(2)

The response tables of the SN ratio and mean for mullite phase percentage are presented in Table 5(a). The response tables provided information about the nature of the process under consideration. The highest difference (based on mean) is classified as the most substantial influence on the desired response. For mullite phase percentage response, the sintering temperature is reported as the most substantial influence parameters, followed by soaking time, POFA composition, molding pressure, and type of POFA in that order. Meanwhile, the response tables of the SN ratio and mean for albite phase percentage are presented in Table 5(b). The most substantial influence parameter for albite phase
percentage response was also reported by the sintering temperature parameter. Differently for further orders, where it is followed by POFA composition, soaking time, molding pressure, and type of POFA.

Table 5. Response table

| Level | a) Mullite phase percentage | b) Albite phase percentage |
|-------|-----------------------------|-----------------------------|
|       | A  | B  | C  | D  | E  | A  | B  | C  | D  | E  |
| SN ratio | 18.528 | 20.285 | 17.590 | 0.000 | 16.121 | -26.60 | -23.56 | -27.51 | -32.64 | -27.33 |
| 2     | 17.766 | 17.630 | 19.743 | 22.351 | 20.680 | -26.65 | -27.11 | -26.93 | -29.17 | -26.07 |
| 3     | 16.525 | 17.743 | 32.091 | 17.642 | 16.121 | -29.21 | -25.43 | -18.07 | -26.48 |        |
| Delta | 0.762 | 3.761 | 1.519 | 32.091 | 4.559 | 0.05 | 5.66 | 2.08 | 14.56 | 1.26 |
| Rank  | 5   | 3   | 4   | 1   | 2   | 5   | 2   | 3   | 1   | 4   |
| Mean  | 19.111 | 22.800 | 17.633 | 1.000 | 15.617 | 27.967 | 19.817 | 29.200 | 43.150 | 29.200 |
| 2     | 19.089 | 16.967 | 20.400 | 15.983 | 24.667 | 27.144 | 28.333 | 26.817 | 31.117 | 23.850 |
| 3     | 17.533 | 19.267 | 40.317 | 17.017 | 9.050 | 34.517 | 26.650 | 8.400 | 29.617 |        |
| Delta | 0.022 | 5.833 | 5.767 | 39.317 | 9.050 | 0.822 | 14.700 | 2.550 | 34.750 | 5.767 |
| Rank  | 5   | 3   | 4   | 1   | 2   | 5   | 2   | 4   | 1   | 3   |

Note: A: Type of POFA; B: POFA composition; C: Molding pressure; D: Sintering temperature; E: Soaking time

Main effect plot for both mullite and albite phase percentage responses are shown in Figure 3(a) and Figure 3(b), respectively. It presents the optimal parameters for both responses by referring main effects plot for SN ratio instead of means. Apparently, the optimal parameters for highest mullite percentage response reported in this condition: 5 wt. % of UGPOFA, pressed at 3-ton, sintered at 1200 °C, and 180 min of soaking time. Meanwhile, for the smallest albite percentage response, optimal parameters reported in this condition: 5 wt. % of UGPOFA, pressed at 4-ton, sintered at 1200 °C, and 180 min of soaking time. Both responses show similar result except for optimal molding pressure. Presently, response table for both response reports that molding pressure as the fourth most substantial parameters. The results significantly for POFA process, where UGPOFA presented as an optimal type of POFA. It actually minimizes the process of POFA by eliminating the grounding and second sieving process. However, this type of POFA apparently considered as the least substantial parameters, where if GPOFA is used, minimum changes are expected.

![Figure 3. Main effects plot for SN ratios of a) Mullite phase and b) Albite phase percentages responses](image)

However, minimum POFA composition was classified for both responses, where it reduces the benefits to the palm oil industry. It still benefited because it might be deals with industry scale levels. The molding pressure for the highest mullite and lowest albite phases percentages are presented by 3 and 4-ton, respectively. Presently, it is classified as insignificant parameters, where the minimum
effect is expected. Differently for the sintering temperature and soaking time, where both responses generate a similar set of optimal parameters. Absolutely, the highest values provided for the optimal sintering temperature because those phases sensitively to the temperature. Meanwhile, the soaking time is provided by the 180 min.

4. Conclusion

The triaxial ceramic employing POFA application was studied by Taguchi design and crystalline phase analyses. The interaction between Taguchi design with quantitative analysis of crystalline phase analysis was mainly focused. The sintering temperature was classified as the most substantial parameters for the formation of mullite and albite phases. It was followed by soaking time, POFA composition, molding pressure, and type of POFA for mullite phase percentage response. Meanwhile, albite phase percentage just twists the position of POFA composition and soaking time in that order. Taguchi design analysis also develops optimal parameters for each objective response. The highest mullite phase percentage was achieved by 5 wt.% of UGPOFA, pressed at 3-ton, sintered at 1200 °C, and 180 min of soaking time. These optimal parameters were also presented by the lowest albite phase percentage, but 4-ton of molding pressure was presented.

It shows the clear interactions developed between Taguchi design and crystalline phase results. The crystalline phase analyses quantitatively presented to provide result regarding numerical results. The amorphous phase recommended included in this study to increase the efficiency of quantitative analysis from XRD plots.

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