Optimization of extrusion process parameter in manufacture of ilmenite TiO₂ reinforced pencil lead

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ABSTRACT

The production of pencil lead with the inclusion of ilmenite for strength improvement and product wear control especially during production was investigated. A Response Surface Methodology (RSM) based on three numeric factors and two center points thus generating sixteen runs was used as tool for the optimization of production process parameters. The major raw materials which are graphite clay and ilmenite were characterized using X-ray fluorescence technique while the surface morphology was studied using scanning Electronic microscopy. The studied input variables were ram pressure, moisture content and die dead angle. Also, an experimental investigation of fracture force and wear rate of pencil lead were carried out. To obtain the optimum values of the properties being investigated, a tribometer and Micro Universal Testing Machine (MUTEM 4) were used for measurements, and response table was generated. The response surface plots showed that all three input variable had considerable impact on the responses. Results showed that the graphite used is made up majorly of carbon and ilmenite (TiO₂). The control factor levels were applied to optimize the desired mechanical properties of reengineered pencil lead using the RSM. Results also showed that with a die angle of 55.3850°ram pressure of 5.716Mpa and moisture content of 22.735, the optimal fracture force is 2.6055N, and optimal wear rate is 0.445 mm/l. These results were validated with those of industrially manufactured ones whose values are 2.95N and 0.35 mm/l respectively. It was concluded that ilmenite can serve as a good additive to pencil lead production.

1. Introduction

Utilization of graphite/clay composite in the production of pencil lead is an old practice, which accounts for over 40% of the world's graphite Pencils (Chelgani et al., 2016; Ying et al., 2014). The idea of incorporating additives provided the improved solution to writing and drawing needs (Iulia et al., 2017). It is these additives that modify and improve the quality and effectiveness of the lead (Alipour et al., 2013). It is evident that legibility and usability is of importance in a typically extruded pencil lead. Structurally the breaking strength of the lead is quite important especially in situation of wood casings that must be sharpened for effective use. The reflectance and surface properties including magnetic performance has also become of need as application of pencil is growing (Kim et al., 2016; Sause 2011).Pencils can be variously classified into types by their: material, use, shape, size, manufacture and length. They can be graded based on the hardness or softness of their leads (Liv and Nakibogu 2016; Kariuki 2012).

Pencil production is chiefly done by extrusion. The product is associated with few imperfections which usually result in some defects. Easily noticed of the defects are; poor compressive strength, high wear rate, low fracture force, surface cracking, chevron cracking, deformed eccentric geometry and non-uniformity in geometry (Popova, 2013).The resulting rod from the extrusion is always bent and irregular in shape. Some other features as grooves and marks on the surface may appear as defects. Non continuous flow of extrusion has its own effect. The extruded materials will come with intermittent supply of compressed materials. The products will end up with some flakes or discontinuities in the lattice formation of the molded materials. From these thin lines, cracks ensure, hence the pencil lead production processes should be optimized to minimize these defects. The grooves and marks may not be fully seen as defect until they are of unacceptable magnitude. A fully smooth lead will as well create a smooth lattice dislike. Adherence to other materials will be a challenge (Abadias 2018).

Properties of the constituting raw materials have their own role to play in the reduction of product failure. The particle size of the granules,
purity of solid minerals and their chemical constituents are some of the factors but not to be presented here (Rutheravan et al., 2016). It is a part of good production requirement to moderate the prevailing operation parameters in the production environment in order to reduce or eliminate these defects. In this work, we examined the effect of moisture content, extrusion pressure and moisture content on the plastically engineered “clay-graphite composite”.

Though product re-engineering form the basic drive to this research, product quality improvement, cost reduction and material availability are also of equal concern. The introduction of ilmenite is a deliberate effort to introduce enough rutile into the product formulae. The needful study is to ascertain the physical, textural and structural behavior of the composite when subjected to the relatively high pressure of the extrusion chamber.

2. Materials and methods

2.1. Materials used for the study

The major materials used for this study are clay, graphite and ilmenite. They were all sourced in their best refined form. The clay used was sourced locally from Uwana in Abia State, ground to a particle size of 90 μm, calcined for purification purposes and void reduction. The Graphite used is a native mineral, and a form of coal. It is plumbago sourced from Saman-Bukonu, kogi State, Nigeria. Ilmenite was sourced from Kuza, Jos Plateau State, Nigeria. Pre-treated to a quality and particle size capable of being utilized. It was used as an additive to clay/graphite. Its inclusion is intended to increase the ease of writing reflection pleochroism and light absorption. The weight composition of these materials used are; 35% clay, 3% ilmenite and 62% graphite.

| Parameter                  | Level |
|---------------------------|-------|
| Die Dead Angle (degree)   | 30    |
|                           | 45    |
|                           | 60    |
| Moisture content (%)      | 15    |
|                           | 20    |
|                           | 25    |
| Pressure (MPa)            | 3.0   |
|                           | 4.5   |
|                           | 6.0   |

Table 1. Parameter and their levels.

Figure 1. Contour and 3D plots of die dead angle vs ram pressure on fracture force.

Figure 2. Contour and 3D plots of die dead angle vs ram pressure on wear rate.
2.2. Manufacturing process

The materials were first dried to eliminate moisture, after which they were ground and sieved into 90μm particle size and properly mixed. Next was the compaction process to form cylindrical shaped blocks ready for extrusion. The direct extrusion method was utilized for the extrusion process at room temperature, after which the extrudite was again dried and gradually baked at an oven temperature of 100 °C.

2.3. Experimental design

2.3.1. Factors and responses

Three process parameters; Die dead angle, moisture content and ram pressure are the selected independent factors to be analyzed against wear rate and fracture force, which form the responses. Three levels of each independent factors were selected for analysis as shown in Table 1, with the aim of minimizing the number of runs. After selection, the Central Composite Design (CCD) tool of the Design Expert software was used to generate the design matrix.

2.3.2. Optimization of process parameters

A second order polynomial of the form shown in Eq. (1), describes the system (Montgomery, 2009).

\[ Y = b_0 + \sum b_i x_i + \sum b_{ij} x_i x_j + \varepsilon \]  

(1)

where y is the response and x is the independent factors.

An important assumption is that the independent variables are continuous and controllable by the experiments with negligible errors.
Figure 4. Optimal solution plots.

Figure 5. A plot of Die angle vs Fracture force.

Figure 6. Variation of fracture force with moisture content.

Table 2. XRF Analysis of ilmenite.

| Component | Unit  | Composition |
|-----------|-------|-------------|
| Na₂O      | Mass %| 0.1183      |
| MgO       | Mass %| 0.2464      |
| Al₂O₃     | Mass %| 6.0470      |
| SiO₂      | Mass %| 10.2321     |
| P₂O₅      | Mass %| 0.0227      |
| SO₃       | Mass %| 0.1899      |
| Cl        | Mass %| 0.955       |
| K₂O       | Mass %| 0.4294      |
| CaO       | Mass %| 0.1284      |
| TiO₂      | Mass %| 47.8505     |
| Cr₂O₃     | Mass %| 0.0895      |
| MnO       | Mass %| 0.4983      |
| Fe₂O₃     | Mass %| 33.7579     |
| NiO       | Mass %| 0.0530      |
| CuO       | Mass %| 0.0107      |
| ZnO       | Mass %| 0.0555      |
| ZrO₂      | Mass %| 0.0206      |
3. Results

3.1. Combined effect of operating parameters on response

The combined effect of the independent variables (moisture content, die dead angle and ram pressure) against the responses (wear rate and fracture force), were studied using contour and surface plots for the purpose of optimizing the output quality.

The effort to obtain the optimal part of safe production gave rise to combined consideration of the factors to the emergent extrusion chamber condition. This condition will give a product output quality, wear rate and fracture force were the system responses considered.

Figure 1 shows the response surface contour plot of fracture force, for the combined effect of ram pressure and die dead angle. A maximum die dead angle and ram pressure of 60° and 6kpa respectively, against a maximum and mean fracture force of 2.6N and 2.5N respectively, were obtained. Also, the ram pressure is seen to increase with decreasing die dead angle from the plot.

Figure 1 shows the contour and 3D response surface plot of die dead angle and ram pressure against fracture force. The response surface plot is an extension of contour plot with one extra coordinate axis. It clearly depicts that the die dead angle influences the fracture force (Rafid, 2014) as well as the ram pressure. The fracture force increases with increasing ram pressure and die dead angle. From the contour plot it can be seen that the region around 6.0MPa ram pressure and 60° die dead angle gives the maximum fracture force.

Figure 2 shows the contour and 3D plots of wear rate for the combined effect of die dead angle and ram pressure. The contour plot shows a maximum die angle of 60°, a maximum ram pressure of 6kpa and minimum wear rate at 0.43 mm/L. The correlation between the design factors and response predicts a quadratic model. The 3D plot clearly shows a desirable low wear rate between 39° and 54° die dead angle as well as 5.4Mpa and 6Mpa ram pressure.

3.2. Optimization of process parameters

The optimization tool of the Design Expert software was employed to optimize the production process parameters of the re-engineered pencil lead. The normal approach which involves selecting the best solution based on economic consideration was adopted and chosen optimal solution presented in Figure 3. By using numerical optimization technique
which is a feature of CCD in the design expect software a combination of factors that concurrently satisfy the requirement placed on the responses and factors could be determined by the software. The impact of each factor on the response was the most important consideration. The over plot of Figure 3 also shows an optimal 55.3848° die dead angle and an X² value of 5.7164 which is the RAM pressure upon a moisture content of 22.7351.

Figures 3a, b, c show plots of die dead angle and RAM pressure for desirability, fracture force and wear rate respectively in search of best points for application.

3.3. Optimal solution plots

Figures 4a, b, c, d and e show optimal combination of the different process parameter which yielded the optimal response. The optimal die dead angle of 55.3848°, 5.7164 kpa ram pressure and 22.7351% moisture content, to give an optimal fracture force and wear rate of 2.6055N and 0.4445 mm/l respectively, were obtained (see Figure 5).

Table 2 shows XRF analysis of ilmenite from Jos Plateau. Ilmenite was introduced as an additive to graphite/clay blend. Ilmenite is made up of 47.8505% of TiO₂ by mass. The clay used also contains up to 1.3976% of TiO₂. Since titanium is widely used in paint manufacture (Idris and Rashan, 2017) and ink industries knowing its resistance to decolouration under ultraviolet light, it makes it suitable for colour retention applications. With its light scattering properties, Ilmenite in pencil lead will brighten the application of lead on paper (Auger and McLaughlin, 2014).

The iron(IV)oxide in ilmenite constituting 33.7579% by mass, is useful in the pigmenting of pencil lead. Since Fe₂O₃ was found in the ilmenite as shown by XRF analysis, the pencil will exhibit some Ferro magnetic properties.

3.4. Product quality

In every production process, the quality of the product is of great importance. To this effect, all the factors that affect the output need due consideration.

3.4.1. Measured values of the systems parameters

Figure 6 shows the variation of fracture force with moisture content at various ram pressures. A linear plot was obtained at 4.5MPa while the maximum fracture force was recorded at 6.0MPa and 20% moisture content. It also shows that the fracture force decreases with an increasing moisture content at 4.5MPa ram pressure.

Figure 7 shows that there exist a linear relationship between wear rate and die angle at 25% moisture content, with 20% moisture content showing the greatest difference in wear rate.

3.5. SEM (Scanning Electron Microscopy)

Figures 8a to d, are SEM images with different compositions and electronic magnifications, at 20.0KV and scale resolution of 20μm and an EDX showing the final product major composition. This studies the surface morphology and the physical properties of pencil lead after extrusion. They show some end of a worn portion but still without cracks or deep scores. At different magnification, the images show a relatively uniform distributed wear which is not necessarily an effect of the systems parameters but the quality of the extrusion technology used. However, the images show that the pencil lead was well compounded. The peaks and valleys could be seen as the product was worked in a production line. The EDX captured Carbon as the major constituent of the lead. This is expected and it follows the design of the product. There are also no injurious element found in the end result as safety of users who may in error eat the lead was put in into consideration. (children use the product).
Figure 10. (a): Solid model of pencil. (b): Deformation on rod. (c): Von mises stress on rod. (d): Elastic strain behavior of the rod.
3.6. X-ray diffraction (XRD) analysis

Figure 9 shows the XRD spectrum of the pencil. The crystalline structure of the sample shows poor crystallinity. At an angle of 39°, the maximum count is 40000 which is low, indicating relatively loose lattice nature. This is desirable since high crystallinity results to low wear rate and poor writing performance (Javaid and Raghunath 2020).

3.7. Structural behavior of the composite

A summary of the structural limits of the product is presented in Table 3, while Figure 10 shows the structural limits of the product. Figure 10(a) is the initial model representing the extruded rod, while Figure 10(b) show a directional deformation. The two ends of the rod experienced the maximal effect of pressure. This is expected since the initial engagement of the lead past with the extrusion die will certainly buildup stresses. The middle is within acceptable range while the load increased at the end. The final deformation rise is associated with the cantilever effect and shear as a result of chop off process. The Von mises stress and elastic strain behavior of the rod shown in Figures 10(c) and 10(d) are within a tolerable yield strength limit. This preliminary test gives a guide to the understanding that the load can withstand sharpening stress as the cutting blade engages the edge of the rod.

4. Conclusion

It is clearly observed the desired strength improvement of this product was achieved by the addition of ilmenite. The structural properties showed appreciable improvement in strength values due its characteristics, notwithstanding some minor production rig imperfections. Worthy of note is the usability of the product as could be seen in the prevalent crystalline nature. This shows that the product will easily write on papers. A validation experiment was performed using an existing pencil brand. The fracture force of the existing pencil brand is 2.95N and a wear rate of 0.35 mm/L (Sause 2014) as compared to the experimental values of 2.605N and 0.445 mm/L respectively. The experimental values closely agree with those of existing brand. The slight difference could result from the capacity of the machine used as well as difference in mixture composition. Hence, extrusion manufacturing method is recommended for pencil lead production.

Declarations

Author contribution statement

Solomon Chuka Nwigbo, Chukwulozie Paul Okolie, JUDE EZECHI DARA, UCHE CLEMENT ATUANYA: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

Data will be made available when manuscript is published by HELIYON.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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