

SOME PHYSICAL FACTORS AND COMPRESSIVE STRENGTH INTERACTIONS IN CELLULOSE ACETATE AGGLOMERATE

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ABSTRACT

Moist cellulose acetate agglomerates were investigated for their compressive strength and some physical factors interactions using statistical methodology. These agglomerates were prepared in a rotating balling disc using different sieve sizes of cellulose acetate powder, with dual purpose kerosene. Experimental process was designed using Two-Level Factorial Design. After obtaining the limits for the experimental process for some of the factors selected, the experimental design was completed using the limits and the design was implemented in producing the cellulose acetate agglomerates. The compressive strength was computed from the damage compressive force obtained during compressive testing as per American Society for Testing and Materials (ASTM) standards. Among the selected factors (particle sizes, agglomerate mass, agglomerate diameter, agglomerate surface area and compressive force), the compressive force reported the highest and positive effect or influence. But agglomerate surface area and agglomerate mass also reported some influence though negative. Particle sizes and agglomerate diameter reported insignificant effect or influence on the compressive strength.

Keywords: Compressive Strength, Cellulose Acetate, Balling Disc, Dual Purpose Kerosene, Compressive Force

1.0 INTRODUCTION

Cellulose Acetate (CA) is the most useful, accessible and advantageous synthetic cellulose ester which is obtained by a chemical process called esterification. Though cellulose is a natural occurring polymer as found in cotton and consists of long chains of anhydro-D-glucopyranose units (AGU) with each cellulose molecule having three hydroxyl groups per AGU except at the terminal ends, CA is obtained by esterifying acetic anhydride with acetic acid in the presence of sulfuric acid (Harika *et al.*, 2012). CA is insoluble in water but soluble in organic solvents. It forms films, hydrophilic, viscosifying and thermoplastic. CA applicability are numerous such as film base for photography, as a component in some coatings, as a material frame for eyeglasses, as a synthetic fiber for cigarette filters, food packages and as a membrane for commercial water purification for military, emergency relief and recreational purposes. CA is also used for production of cellulose beads which are used for immobilization of enzymes, as specific absorbents for controlled releasing of active pharmaceutical ingredient and as a medium for separation (Fischer *et al.*, 2008).

Agglomeration is an important process used in many industries to improve the characteristic of a material, giving benefits to the end users in the form of improved quality and function of the final product. Agglomerates are created by adding particles, small quantities at a time, about a central tetrahedral arrangement of primary particles (Capes & Sutherland, 1967). The agglomeration mechanisms achieve adhesive and cohesive bonding during the process operations to produce agglomerates. The surface tension of the bridging liquid is the primary source of the magnitude of these forces. The agglomerates are affected by the moisture content, size of the powder fine particles and structure of the agglomerate. The study of agglomerate strength is of vital importance in several industrial applications such as pharmaceutical, chemical (detergent), process and food industries.

Due to its applications, experimental findings on agglomerate strength are not consistent with theoretical models on effects of binder and CA physical characteristics on the crushing strength (Haines, 1925). In an attempt to investigate this interaction, another approach is presented by evaluating the interaction effects of some physical characteristics of the CA agglomerate. The crushing strength is measured by compression test and Two-Level Factorial analysis is performed to estimate the physical characteristics of CA agglomerate that have the highest contribution to the CA agglomerate strength. This strength value is critical when designing CA agglomerates operations to prevent damage and subsequent failure.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials used for this study are cellulose acetate, water, dual purpose kerosene and alcohol. The instruments used are vernier caliper, weighing scale and three different sieves. The equipment used are mechanical sieve shaker, balling disc and Universal Testing Machine.

2.2 Design of Experiment

The experiment was designed using a Two-Level Factorial technique which obtained sixteen (16) runs using five (5) factors for this investigation. The five (5) factors are sieve sizes, agglomerate mass, agglomerate diameter, agglomerate surface area and compressive force (L).

The compressive strength was evaluated from fundamental load and area relationship. The crushing force was obtained from crushing the agglomerate and the strength evaluated with the following relationship:

$$\text{Compressive Strength (CS)} = \frac{\text{Crushing Load (L)}}{\text{Agglomerate Surface Area}} \quad (1)$$

Since the shape of the final shaped agglomerate is a sphere, the surface area of a sphere is applied.

$$\text{Surface Area of a Sphere} = 4\pi r^2. \quad (2)$$

$$\text{From equation 1 and 2, a relationship is derived as } L = CS\pi D^2. \quad (3)$$

2.3 Method

Cellulose acetate powder was placed in a mechanical sieve shaker having three (3) different sieve sizes of 0.354 mm, 0.42 mm and 0.5 mm. The powder was then separated by the different sieves by the mechanical shaker which stopped when no powder was passing through the sieves anymore. The powder left on each sieve was collected and stored separately.

Next, the binder (dual purpose kerosene) was stored in an aero spray container. As the balling disc rotated at a steady speed, cellulose acetate powder was feed into the balling disc while the binder was sprayed into the rotating balling disc. The binder spraying was well controlled to allow the cellulose acetate powder to agglomerate. Having formed the required agglomerate structure, the agglomerate spheres are removed from the balling disc; the agglomerate diameter was measured using vernier caliper in three directions: x, y and z and the average of the measured diameter is recorded as the agglomerate diameter. The agglomerate mass was measured using a weighing balance. The agglomerate was placed in the Universal Testing Machine and the crushing load or compressive force was determined (Ciftcioglu *et al.*, 1986).

3.0 RESULTS AND DISCUSSION

The compressive test results are presented in Table 3.1

Table 3.1 Experimental results obtained for the various factors and response.

S/N	Material/Sieve Sizes (mm)	Mass of CA Powder used (g)	Diameter of CA agglomerate formed (mm)	Area of CA agglomerate formed (mm ²)	Compressive Force (N)	Compressive Strength (N/mm ²)
1	0.50	43	40	502.7	0.734	1.46
2	0.42	37	39	490.2	0.926	1.89
3	0.35	25	32	402.2	0.833	2.07

The Two-Level Factorial Design is given below and the various plots showing the factors interaction and influence on the compressive strength also displayed and discussed.

Table 3.2 Half factorial design of the various factors and response

Run	Factor 1: Mass (g)	Factor 2: Diameter (mm)	Factor 3: Area (mm ²)	Factor 4: Sieve Sizes (mm)	Factor 5: Compressive Force (N)	Response 1: Compressive Strength (N/mm ²)
1	25	40	402.2	0.35	926	2.3
2	43	32	502.7	0.35	926	1.84
3	43	40	402.2	0.5	734	1.83
4	43	40	502.7	0.5	926	1.84
5	43	40	502.7	0.5	926	1.84
6	25	32	402.2	0.5	926	2.3
7	25	32	502.7	0.5	734	1.46
8	43	32	502.7	0.35	926	1.84
9	25	32	502.7	0.5	734	1.46
10	25	40	502.7	0.35	734	1.46
11	43	32	402.2	0.35	734	1.83
12	43	32	402.2	0.35	734	1.83
13	25	40	402.2	0.35	926	2.3
14	25	32	402.2	0.5	926	2.3
15	25	40	502.7	0.35	734	1.46
16	43	40	402.2	0.5	734	1.83

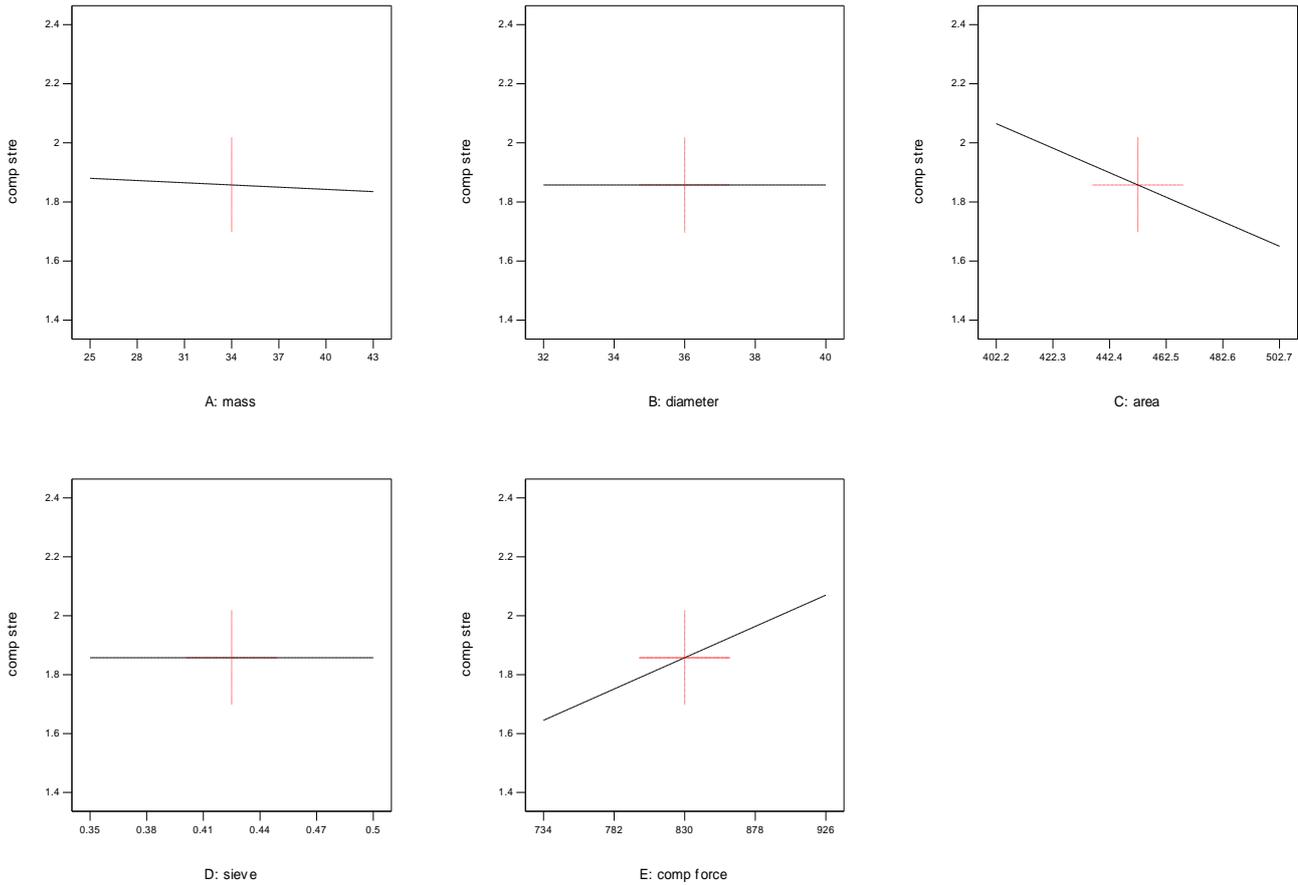


Figure 3.1 Effects of individual factors plots against the response

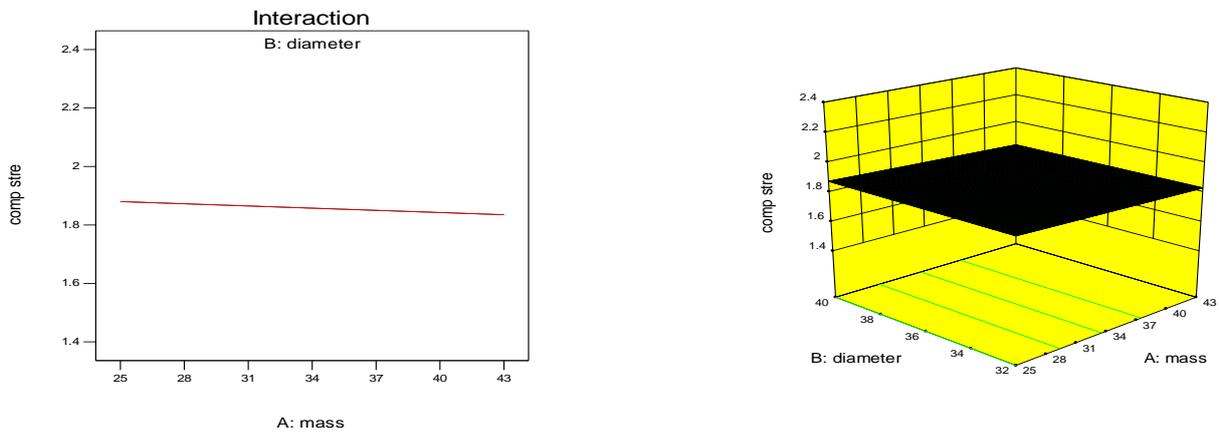


Figure 3.2 Interaction and 3D surface plots of mass and diameter against compressive strength

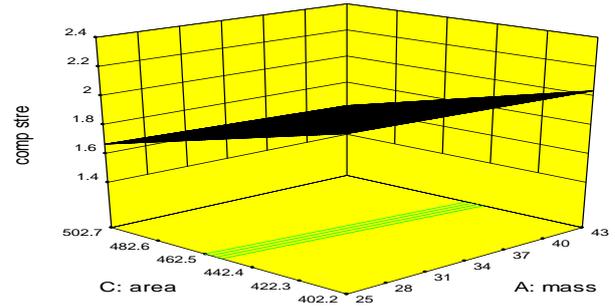
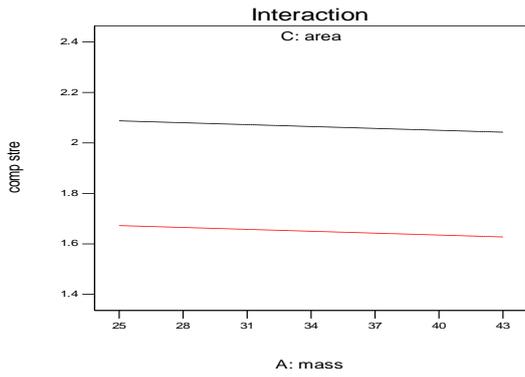


Figure 3.3 Interaction and 3D surface plots of mass and area against compressive strength

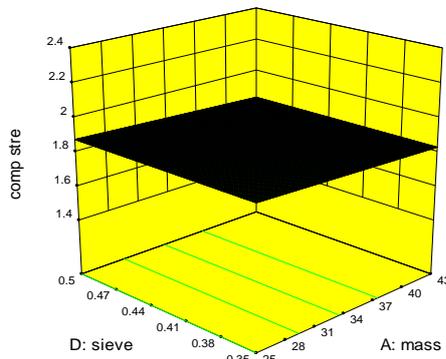
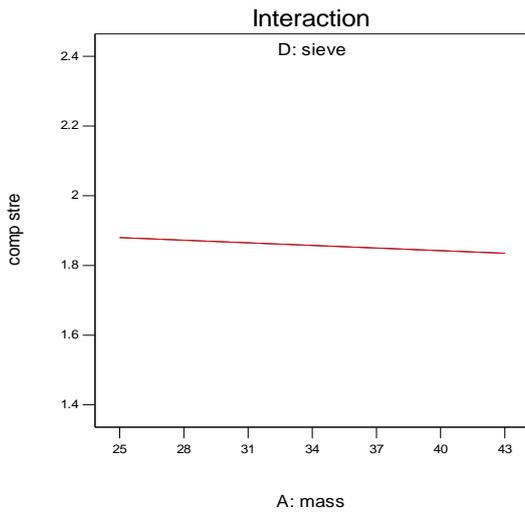


Figure 3.4 Interaction and 3D surface plots of mass and sieve sizes against compressive strength

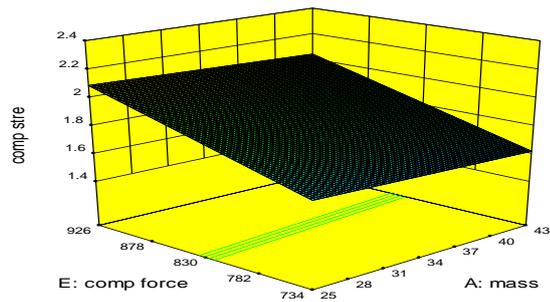
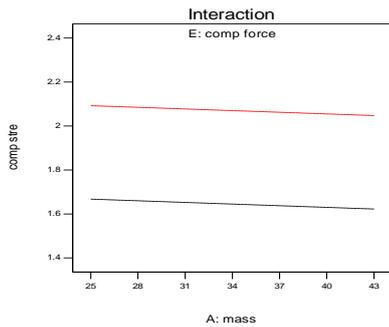


Figure 3.5 Interaction and 3D surface plots of mass and compressive force against compressive strength

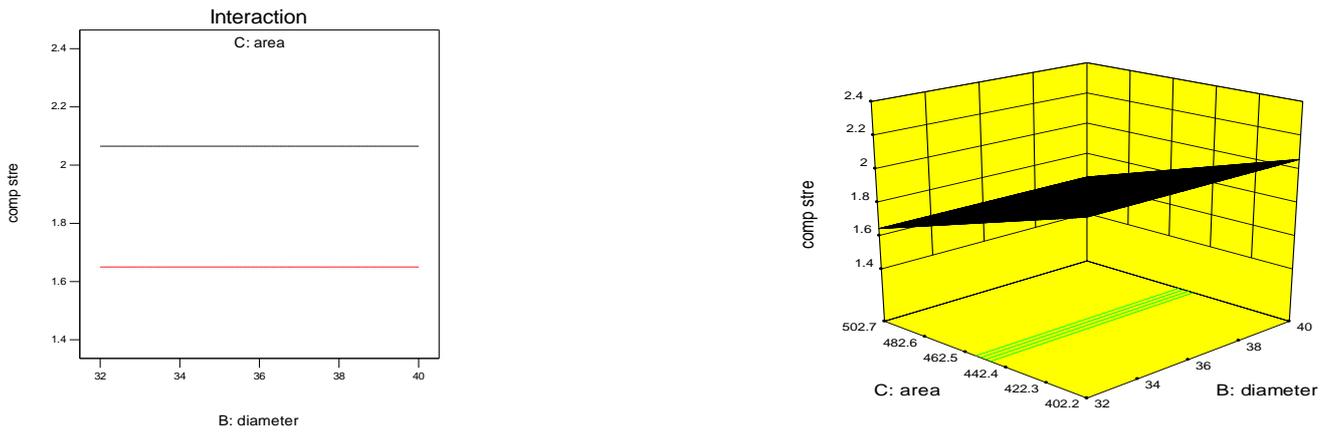


Figure 3.6 Interaction and 3D surface plots of diameter and area against compressive strength

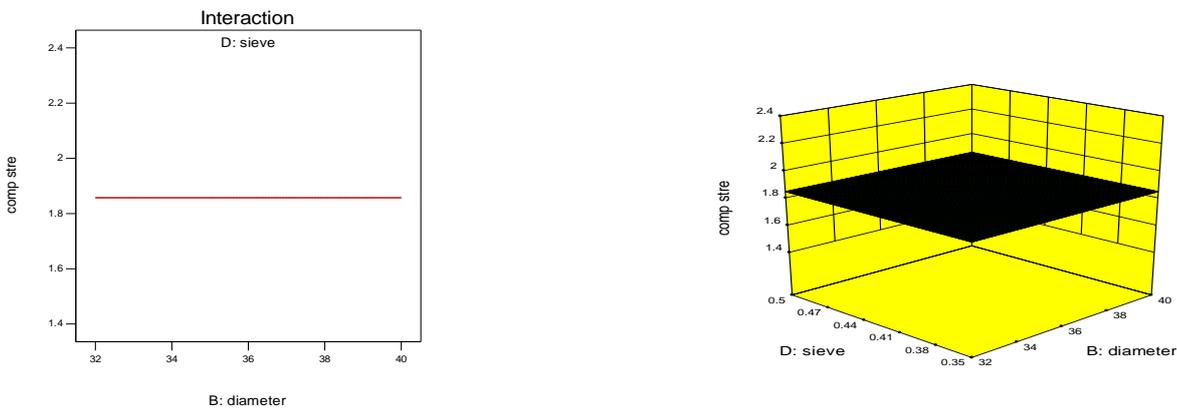


Figure 3.7 Interaction and 3D surface plots of diameter and sieve sizes against compressive strength

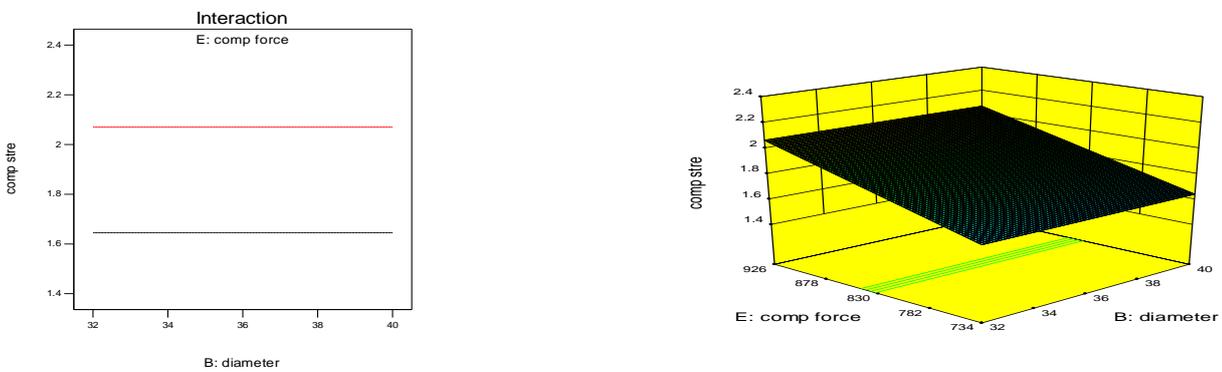


Figure 3.8 Interaction and 3D surface plots of diameter and compressive force against compressive strength

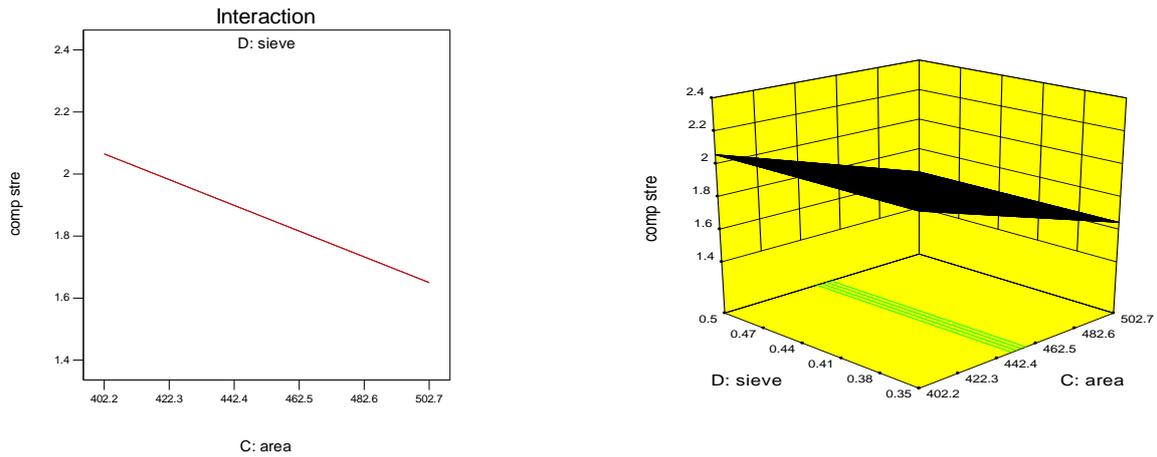


Figure 3.9 Interaction and 3D surface plots of area and sieve size against compressive strength

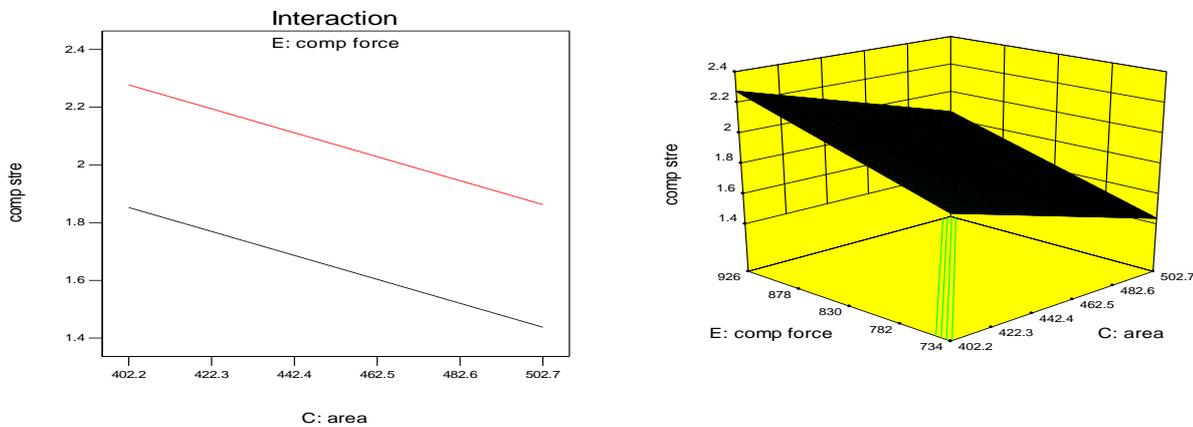


Figure 3.10 Interaction and 3D surface plots of area and compressive force against compressive strength

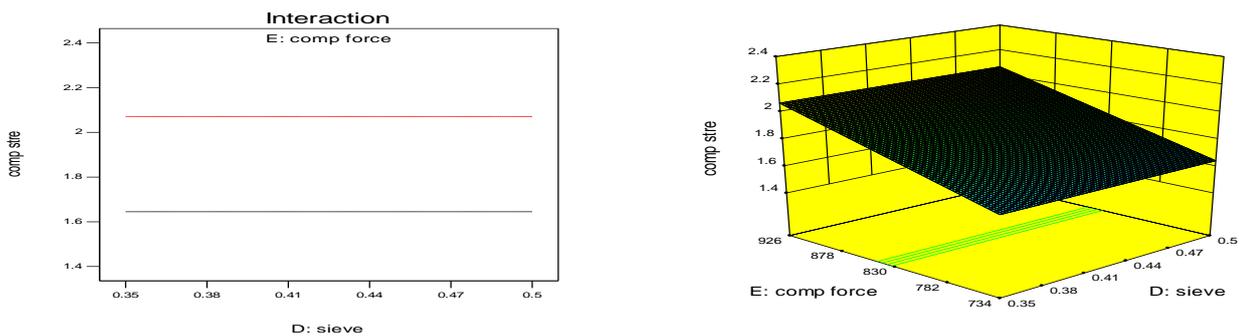


Figure 3.11 Interaction and 3D surface plots of sieve size and compressive force against compressive strength

Table 3.1 tabulates the factor values (sieve sizes, agglomerate mass, agglomerate diameter, agglomerate surface area and compressive force) and response (compressive strength) obtained during experimentation and Table 3.2 provides the half factorial design obtained after inputting the factors limiting values and response values (Goos & Gilmour, 2013; Goos & Steven, 2017).

Figure 3.1 depicts the effects of individual factors on the response. It shows that agglomerate diameter and sieve sizes (CA powder particle sizes) factors show insignificant effect or influence on the compressive strength. But equation 2.3 implies that agglomerate diameter should have influence or effect on the

agglomerate compressive strength by the power of two. The anomaly might be from the agglomerate diameter measurement which contains the error largely interpreted by the plot. The particle size insignificant effect or influence might be from the powder sizes measurement and low powder surface area binder adhesion and cohesion (Subero *et al.*, 1999). The mass of the CA agglomerate showed positive influence on the compressive strength; whereby increase in agglomerate mass decreased the compressive strength. This trend can be linked to the powder density of the CA powder activated due to the agglomerate binder. The surface area of the CA agglomerate showed significant positive influence on the compressive strength. There was visible positive effect by the surface area but as it increases continually, compressive strength decreased. The compressive force showed a positive influence on CA agglomerate. A steady positive increase was recorded by the compressive force and it resulted to a steady increase of the compressive strength proportional to the compressive force.

Figure 3.2 showed the interaction and 3D surface plot of agglomerate mass and agglomerate diameter against compressive strength. There was low interaction by agglomerate mass and agglomerate diameter factors on the compressive strength with the agglomerate mass showing higher influence on the compressive strength on the 3D surface plot. Figure 3.3 showed the interaction by agglomerate mass and surface area factors on the compressive strength. The interaction plot showed the surface area higher than the agglomerate mass and the 3D surface plot depicts the same trend. Figure 3.4 showed low interaction by agglomerate mass and sieve sizes on compressive strength. The 3D surface plot showed the agglomerate mass having higher significant influence on the compressive strength. Figure 3.5 showed the interaction of agglomerate mass and compressive force on the compressive strength. The compressive force is higher in its effect than the agglomerate mass and the 3D surface plot revealed the interaction in the compressive force factor path which also had higher influence.

Figure 3.6 depicts the interaction of agglomerate diameter and surface area on the compressive strength. The surface area has higher interaction effect than agglomerate diameter and the 3D surface plot showed the interaction in the path of the surface area factor. Figure 3.7 showed no interaction by agglomerate diameter and sieve sizes on the compressive strength. This is in line with the flat 3D surface plot. Figure 3.8 showed that compressive force has higher influence than agglomerate diameter. The 3D surface plot revealed that compressive force factor caused most of the effect and have the interaction path. Figure 3.9 depicts the interaction of surface area and sieve sizes on the compressive strength. The surface area factor produced most of the influence on the compressive strength and 3D surface plot reports the surface area factor having the interaction path. Figure 3.10 depicts the interaction of surface area and compressive force factors on the compressive strength. The compressive force is higher on the interaction plot and the 3D surface plot revealed a diagonal interaction path for both factors with compressive force factor having a little more of the interaction path. A shared interaction by both factors revealed vivid interaction by both factors. Figure 3.11 showed the interaction of compressive force and sieve sizes factors on the compressive strength response. The compressive force factor had higher influence than sieve sizes factor and principally had the significant effect on the compressive strength. The 3D surface plot revealed the interaction path as the compelling effect on the compressive strength.

4.0 CONCLUSION

This study determined the compressive strength and some factors interactions on the compressive strength of moist CA agglomerates. The study noted that agglomerate diameter and sieve sizes (particle sizes) factors show insignificant effect or contribute very low to the compressive strength of the moist CA agglomerates response and also do not interact quantitatively with any other factors. Therefore, they may be said to be passive factors. Their passivity are characterized by the powder low bonding attributes and precise parameter mensuration. However, agglomerate surface area, agglomerate mass and compressive force factors showed different levels of influence on the compressive strength and interacted with each factors significantly and

contributing to the compressive strength of the moist CA agglomerates. Conclusions can be drawn from literature that each factor (agglomerate mass, agglomerate surface area and compressive force) possess certain properties which attribute to their contributions or influences or effects in the compressive strength of the moist CA agglomerates. The agglomerate mass has a bulk resistance which is attributed to the CA powders held together by the binder or bridging liquid by cohesion and adhesion forces. Most of the resistance comes from internal bonding energies occasioned by cohesion and adhesion forces (Omenyi, 1978; Omenyi *et al.*, 1998; Tosello *et al.*, 2019). The agglomerate surface area is the periphery of the moist CA agglomerates. The resistance here is at the surface of the moist CA agglomerates held together by stronger cohesion and adhesion forces than within the moist CA agglomerates. Pores and voids on the agglomerate surface also contribute to the surface resistance and subsequently the compressive strength. It was favourable noted that compressive force factor increased in the same proportion of the compressive strength. When high compressive strength of moist CA agglomerates is desired, this study revealed that the most contributing factors are the agglomerate mass and agglomerate surface area which is necessitated by the type of agglomerate formation process (Wynnyckyj, 1985).

REFERENCES

- Capes, C. E., & Sutherland, J. P. (1967). Formation of Spheres from Finely Divided Solids in Liquid Suspension. *I&EC Process Design and Development, American Chemical Society*, 6, 146.
- Ciftcioglu, M., Akinc, M., & Burkhart, L. (1986). Measurement of Agglomerate Strength Distributions in Agglomerated Powders. *American Ceramic Society Bulletin*, 65(12), 1591–1596.
- Fischer, S., Thummler, K., Volkert, B., Hettrich, K., Schmidt, I., & Fischer, K. (2008). Properties and Applications of Cellulose Acetate. *Macromolecular Symposia*, 89–96.
- Goos, P., & Gilmour, S. G. (2013). *Testing for Lack of Fit of Blocking and Split Plot Response Design*.
- Goos, P., & Steven, G. (2017). Testing for lack of fit in blocked, split-plot, and other multi-stratum designs. *Journal of Quality Technology*, 49(4), 320–336. <https://doi.org/10.1080/00224065.2017.11918000>
- Haines, W. B. (1925). Studies in the Physical Properties of Soils. *The Journal of Agricultural Science*, 15, 529–535.
- Harika, K., Sunitha, K., Pavan, K. P., Maheshwar, K., & Madhusudan, R. Y. (2012). Basic Concepts of Cellulose Polymers - A Comprehensive Review. *Archives of Pharmacy Practice*, 3(3), 202–216.
- Omenyi, S. N. (1978). *Attraction and Repulsion of Particles by Solidifying Melts*. University of Toronto, Canada.
- Omenyi, S. N., Okeke, B., & Capes, C. E. (1998). Physico-chemical Parameters Determining the Strength of Moist Agglomerates. *Journal of Engineering Research*, 6(2), 0795–2333.
- Subero, J., Ning, Z., Ghadiri, M., & Thornton, C. (1999). Effect of interface energy on the impact strength of agglomerates. *Powder Technology*, 105(1–3), 66–73. [https://doi.org/10.1016/S0032-5910\(99\)00119-9](https://doi.org/10.1016/S0032-5910(99)00119-9)
- Tosello, G., Gulcur, M., Whiteside, B., Coates, P., Luca, A., De Sousa Lia Fook, P. V., Riemer, O., Danilov, I., Zanjani, M. Y., Hackert-Oschätzchen, M., Schubert, A., Baruffi, F., Achour, S. Ben, Calaon, M., Nielsen, C. V., Bissacco, G., Cannella, E., Rasmussen, A., Bellotti, M., ... Zeidler, H. (2019). Micro product and process fingerprints for zero-defect net-shape micromanufacturing. *European Society for Precision Engineering and Nanotechnology, Conference Proceedings - 19th International Conference and Exhibition, EUSPEN 2019*.
- Wynnyckyj, J. R. (1985). Coorelation between the Strength Factor and the True Strength of Agglomerate Spheres. *The Canadian Journal of Chemical Engineering*, 63.