Effect of metallic fillers on the hardness of polystyrene composites: An experimental investigation

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Abstract. In this study, the effects of composite thickness (2.5-7.5 mm), filler content (15-45%w/w) and particle sizes (20-100 µm) on the hardness of Polystyrene metal composites were examined. The metals comparatively considered were aluminium particles and iron fillings. It was observed that smaller metal particle sizes in the composite matrix and greater filler proportions will lead to improvement in hardness. Composite thickness did not have a significant effect on the hardness of the metal composites. PS-Al composite is harder than PS-Fe composite with the optimal hardness being 43.7 and 11 respectively. The factor levels that will optimise hardness for PS-Al composite are thickness of 7.5 mm, filler of 39% and particle size of 20 µm. For PS-Fe composites they are thickness of 5.9 mm, filler of 45% and particle size of 34µm. The metallic composites (PS-Al and PS-Fe) exhibited contrasting mechanical strength when viewed under SEM with a homogenous micro-structure of high interfacial interaction between the metal particles (Aluminium) and the PS resin matrix while the PS-Fe metallic composite under micro-structural analysis displayed relatively moderate to low tensile strength and hardness with weak interfacial interaction between the Iron particles and the PS resin.

Keywords: Polystyrene, Aluminium, Iron, Composite, Metal, Response surface

1. Introduction
Materials with dynamic hardness and tensile strengths can be combined on a microscopic scale in alloying of metals, organic compound-metal matrices or other forms resulting in material of distinctive properties, macroscopically homogenous for all practical purposes [1]. The toughness of composites material is governed by the stress-strain relation that is valid for conventional materials [2]. Applications of composite materials play essential roles in engineering fabrications such as structural and functional materials, scientific fields as nanocomposites and in medicine for active organic ligands [11].
Research in polymer composites has generated wider area of interests for more than half a century as a result of the rising demand for materials with a specific blend of properties with high structural and...
flexural strength. A unique set of properties is developed in the composites, such as enhanced mechanical properties, electrical conductivity, thermal conductivity, gas barrier properties and so on, lacking in naturally-occurring materials, is accomplished in the category of composites [3], [4]. Metals are high value products but with some related environmental issues associated with its residual wastes; metallic fillings and chips from in use and operations on iron, aluminium are non-degradable, which makes recycling a vital alternative. Utilization of recycled metals such as (Iron) Fe, (Aluminium) Al which ordinarily are regarded as waste saves energy when compared to extraction from its ore while landfills are saved [11, 12] [5]. The graded metals (Al, Fe) of various particle sizes thoroughly dispersed in its polystyrene based resin (PBR) matrix are considered in this study [6]. PBR was previously used as matrix in the production of wood plastic composites [11]. Metallic powder had been chosen due to their 100 % recyclability, readily available, non-degradable as solid waste to avoid land pollution, high specific strength and modulus [7]. Composites materials of desirable qualities employ the utilization of large scale materials, efficient machinery and thorough method of blending and re-composition. [8]. The conductive properties of these composites is recently receiving novel researches and experiments as promising source of alternative to metals and alloys due to their significant reduction in pitch, weight and volume and increased environmental compatibility. This work is focused on elucidating the effects of factors like filler particle size, filler concentration and composite thickness on the hardness of the PS-Al and PS-Fe composites, and optimising these factors with the aid of response surface methodology.

2. Materials and Methods

2.1. Materials
Polystyrene polymer is sourced from disposed electronic packaging, gifts items packing and other products which require the need for packing at various shops and outlets. Metallic fillers sourced in form of fillings from metals and aluminium fabricators (Al, Fe) washed in water (To remove impurities), dried and classified to sizes of 20 mm, 60 mm and 100 mm.

2.2. Preparation of polystyrene-metal composites
The composites (PS-Metal) were produced through manual blending and mixing of metal and PBR. Recycled Polystyrene was dissolved in the prepared petroleum solvent at room temperature to form viscous polystyrene based resin (PBR) as previously reported [11, 12]. A calculated percentage of PBR- metallic fillers (15%, 30% and 45%) relative to the PBR were measured and mixed together thoroughly in a container at low speed for 10 minutes to obtain a homogenous 100% composite. The mixture of the matrix material was then poured into the mould with dimension 20 cm x 8 cm x 1 cm to obtain the metallic-polymer composite which was cut in to different dimensions and allowed to cure for 3 days at room temperature. Various mixes were prepared according to the design of experiments.

2.3. Hardness test
The Vickers indenter is a square based pyramidal-shaped diamond indenter with face angles of 136°. The Vickers hardness was measured based upon the force divided by the surface area of the indentation of the metallic composites samples. The hardness of the (PS-Metal) composites were measured after polishing to a 1 mm finish. The Brinell hardness values of the samples was measured using LECO micro hardness tester with model no.: LM700AT to obtain an indentation which would be representative of the macrostructure of the material. The material hardness of the samples is calculated by using equation (1):

$$HV = \frac{1854.4F}{d^2}\left[9\right]$$

(1)
In order to eliminate possible segregation effects; the mean of three tests was taken for each specimen.

2.4. SEM analysis

The scanning electron microscope (SEM) was used to identify morphology of the metallic composite samples (PS-Al and PS-Fe) under four different resolutions. The surfaces of the composite specimens were examined directly by scanning electron microscope. The samples will be washed, thoroughly cleaned, air-dry and coat with 100 Å thick platinum in JEOL sputter ion coater and then observed (SEM) at 20 kV.

2.5. RSM Optimisation

The factors considered and the ranges studied were composite thickness (2.5-7.5 mm), filler content (15-45%) and particle sizes (20-100µm). The optimisation experiment was prepared according to response surface methodology and 3-factorial design with the aid of Design expert 10.0. The software was also used to develop response surface models, analysis of variance (ANOVA) and response plots.

3. Results and discussion

The first category of plastic-metal composites was obtained from polystyrene resins and aluminium particles (to be denoted as PS-Al) while the second category was obtained using iron filings (to be denoted as PS-Fe). The factors considered were: composite thickness (2.5-7.5 mm), filler content (15-45%) and particle sizes (20-100 µm). After obtaining the results based on the design of experiments (DOE), the results are presented in Table 1 and the findings are discussed in the next subsections.

Table 1. Hardness test result for PS-Al and PS-Fe composites at 490.3mN and 10sec

| Samples | Average Vickers (PS-Al) | Average Vickers (PS-Fe) |
|---------|------------------------|------------------------|
| 1       | 4.7                    | 6.3                    |
| 2       | 2.6                    | 5.5                    |
| 3       | 2.3                    | 5.7                    |
| 4       | 6.7                    | 6.6                    |
| 5       | 2.7                    | 4.4                    |
| 6       | 3.2                    | 4.5                    |
| 7       | 6.8                    | 3.6                    |
| 8       | 9.4                    | 11.7                   |
| 9       | 7.4                    | 7                      |
| 10      | 6.7                    | 16.8                   |
| 11      | 4.5                    | 6.8                    |
| 12      | 5.6                    | 9.1                    |
| 13      | 6.8                    | 13.5                   |
| 14      | 7.2                    | 5.4                    |
| 15      | 3.9                    | 5.7                    |
3.1. Effect of factors on PS-metal composites

The effect of composite thickness, filler content and particle sizes on the hardness of polystyrene-aluminium (PS-Al) and polystyrene-iron (PS-Fe) composites are examined in this section. Response surface plots will be used to elucidate the effect and interactions of these factors. It will be initially put forward stated that hardness is a surface property of the composites and the % amount of metal fillers alongside the particle sizes of the metals used in the composite development is expected to be the only major factor to affect this property. However, there are secondary factors that indirectly affect the hardness of the composites. They include the extent of curing of the composite (alongside the curing technique utilised), the extent of cohesion and metal-resin miscibility (alongside the metal affinity for the specific resin) and the nature of the metals being used. The addition of a third factor which is the thickness of the composite helps to capture the effect of curing on the hardness of the composites as thinner composites will cure faster than thicker ones. On this premise, the effects of the factors were studied.

3.1.1. Effect of Thickness and filler content on the hardness of PS-Al composites

From Figure 1, it’s observed that there is only a slight effect of filler content and composite thickness on the hardness of the derived composites. Within the range of the filler content studied, hardness increases slightly with increased fillers % up to a maximum then drops gently beyond. This is expected because the higher the metals in the composite the harder the surface are expected to be. However, the drop in hardness beyond the optimal threshold is a result of reduced interfacial interaction between the metal particles and the PS resin matrix. This weak cohesion between filler and resin generally weakens the composite and have been shown to negatively impacts virtually all mechanical properties of the composite (of which hardness is inclusive) [10].
The relationship between filler content and hardness holds for all domain of thickness. The thickness of the composite displays a barely noticeable effect on the hardness of its surface. Only at the highest thickness studied was a slight improvement in hardness noticed. Since thicker composites will take longer to cure, we can infer that a slower curing rate has a positive effect on the surface hardness. This relationship is not quite explicit and further studies will be needed in this regard to tell us more.

3.1.2. Effect of thickness and particle size on the hardness of PS-Al composites

Figure 2 reveals that smaller particle sizes of the metal fillers will result in harder composites. This relationship holds for all domain of composite thickness. We can infer that the smaller particles sizes promote surface cohesion, miscibility and blending between the metal particles and the polystyrene resin. Consequently, most mechanical properties are improved in composites where the resins and fillers have very strong surface interactions (and hardness is not an exception).
3.1.3 Effect of Filler content and particle size on the hardness of PS-Al composites

From Figure 3, it’s observed that the earlier elucidated hardness-particle size relationship still holds for all domain of filler content. However, for large particle sizes, changes in the quantity of filler have little effect on the hardness. For smaller particle size, the amount of filler in the composite then has a more significant effect. At that range, lower filler content favours the hardness of the resin up to a threshold.

![Figure 3. Response surface plot of the effect of filler content and particle size on the hardness of PS-Al composites](image)

3.1.4 Effect of Thickness and filler content on the hardness of PS-Fe composites

After examining the effect of these 3 factors on polystyrene-aluminium (PS-Al) composites; we will proceed to look at polystyrene-iron (PS-Fe) composites. Firstly, examining the vertical axis, it will be observed that the effect of these two factors on the hardness is only marginal and for very close values of factors, the general trend may not be identifiable (especially for thickness). Increasing filler content increases the hardness of the composite and this holds for all domain of thickness. There is also a threshold of optimal composite thickness beyond which increasing the thickness does not affect the hardness anymore. It should be noted that this is a very marginal effect that may not be noticeable at very close range of thickness.
3.1.5 Effect of Thickness and particle size on the hardness of PS-Al composites

Figure 5 is a quite interesting plot as it reveals a major combinatorial effect of particle size and composite thickness on the hardness of the composite. For composites that are not thick, there is little effect of particle size on the hardness. For thicker composites, smaller particle size will lead to composites with harder surfaces and vice-versa. Further studies are recommended to understand why this is so.

Figure 4. Response surface plot of the effect of Thickness and filler content on the hardness of PS-Fe composites

Figure 5. Response surface plot of the effect of Thickness and particle size on the hardness of PS-Fe composites
3.1.6 Effect of Filler content and particle size on the hardness of PS-Al composites
Smaller particle size will give harder PS-Fe composites. This holds for all domain of filler content. As earlier said, smaller particles sizes promote surface cohesion, miscibility and blending between the metal particles and the resin which helps to improve the hardness. Since the hardness is an index of indentation, the well mixed and homogenised composite will tend to resist indentation more effectively.

![Figure 6](image_url)

**Figure 6.** Response surface plot of the effect of filler content and particle size on the hardness of PS-Fe composites

3.1.7 Summary and comparison of factor effects for both classes of metal composites
3.1.7.1 Particle size
For both PS-Al and PS-Fe composites, lower particle size gives harder composites. However, the domain of optimality is sharper for PS-Al than for PS-Fe composites. Also (if we consider the y-axis of both plots) we will observe that PS-Al composite is harder than PS-Fe composites. This helps us to make a very important inference. Aluminium has a far better affinity for polystyrene than iron. Though iron is a naturally harder material than aluminium, its lack of affinity for the PS resins results in composites with relatively much susceptibility to indentation due to lesser homogeneity between the two major constituents of the material.

![Figures 7(a-b)](image_url)

**Figures 7(a-b).** Effect of particle size on hardness for metal composites
3.1.7.2 Filler content
A greater proportion of metal in the composite is expected to make it harder hence the trends observed in figures 8a and 8b is not surprising. However, PS-Al composite shows maxima while Ps-Fe doesn’t. For a well homogenised composite (PS-Al), there will always be a threshold beyond with the excessively high amount of metal fillers in the composite will negatively impact the cohesion of the particles and the resins which will consequently lead to reduction in hardness. For a composite with a loss less homogeneity (PS-Fe), this effect will not be apparent as the resultant hardness in the first place was not due to excellent cohesion of particles with resin. If we consider the y-axis of both plots we will also observe that PS-Al composite is harder than PS-Fe composites (irrespective of the fact that the iron metal is naturally harder than the aluminium metal).

![Figures 8(a-b). Effect of filler size on hardness for metal composites](image)

3.1.7.3 Composite thickness
Figure 9a and 9b is the final confirmation of the distinctions between both composites. Ideally, thickness of the developed composite material is not supposed to have a significant effect on the hardness of the composite which is an index of surface indentation. This is observed for the case of PS-Al composite. However, a trend and maxima is observed for PS-Fe composite. Further studies will be needed to understand why an optimum thickness is observed.

![Figures 9(a-b). Effect of composite thickness on hardness for metal composites](image)
3.2 Numerical Optimisation

Design expert 10.0 was used to perform a numerical optimisation of the studied factors to determine the best combination that will yield the hardest materials. As presented in Table 2, the range for optimisation of the factors was the same as those used for the study itself. The goal in both cases was to maximise the hardness while other factors were kept in range.

Table 2. Summary of optimisation results

| Metal Composite | Optimisation Goals                          | Optimisation results |
|-----------------|---------------------------------------------|----------------------|
| PS-Al           | All factors kept in range studied, maximise hardness | Thickness (mm): 7.5, Filler (%): 39, P. Size (µm): 20, Removal eff (%): 43.7 |
| PS-Fe           | All factors kept in range studied, maximise hardness | Thickness (mm): 5.9, Filler (%): 45, P. Size (µm): 34, Removal eff (%): 11 |

The optimal hardness of PS-Al and PS-Fe composites are 43.7 and 11 respectively. Complementing the earlier observations, PS-Al composite is harder than PS-Fe composite. From the optimal factors it’s observed that the highest thickness studies (7.5 mm) was optimal for PS-Al while the optimal thickness for PS-Fe is 5.9 mm. The optimal filler content for PS-Al was 39% while the highest filler content studied (45%) was optimal for PS-Fe. The minimum particle size studied (20 µm) was optimal for PS-Al while the optimal particle size for PS-Fe was 34µm.

3.3 Response surface modelling

One of the reasons for modelling is to develop empirical correlations that can predict the hardness of the composite given known levels of the various factors. It can serve as a good estimator in cases where cost, profitability, feasibility and preliminary analysis are being conducted. This can then serve as a prelude before laboratory trials, development of prototypes and pilot studies can commence.

3.3.1 Modelling for PS-Al composites

The software predicted a quadratic model as the best fit of the experimental data. The final model equation in terms of actual factors is presented in equation 2

\[
\text{Hardness (Al)} = 3.042 - 5.34A + 3.257B - 0.8887C + 0.016AB - 0.0186AC - 0.0091BC + 0.6376A^2 - 0.041B^2 + 0.0079C^2
\]  

(2)

Where A is Thickness in mm, B is filler content in percentage and C is particle size in µm. From Table 3, it is observed that the model is significant (p<0.05). This means that the model is suitable for making estimations of hardness based on these factors. It is also observed that changes in the size of the metallic particle is the most significant factor that affects the hardness of the composite.
### Table 3. ANOVA Table for PS-Al Response surface model

| Source           | Sum Squares | Df | Mean Square | F Value | p-value Prob > F |
|------------------|-------------|----|-------------|---------|------------------|
| Model            | 5319.46     | 9  | 591.05      | 4.39    | 0.0022 Significant |
| A-Thickness      | 18.00       | 1  | 18.00       | 0.13    | 0.7182           |
| B-Filler Content | 447.00      | 1  | 447.00      | 3.32    | 0.0822           |
| C-Particle size  | 2802.51     | 1  | 2802.51     | 20.80   | 0.0002           |
| AB               | 4.32        | 1  | 4.32        | 0.032   | 0.8595           |
| AC               | 41.44       | 1  | 41.44       | 0.31    | 0.5848           |
| BC               | 354.25      | 1  | 354.25      | 2.63    | 0.1192           |
| A²               | 113.60      | 1  | 113.60      | 0.84    | 0.3685           |
| B²               | 609.68      | 1  | 609.68      | 4.53    | 0.0449           |
| C²               | 1127.04     | 1  | 1127.04     | 8.36    | 0.0085           |
| Residual         | 2964.13     | 22 | 134.73      |         |                  |
| Lack of Fit      | 2964.13     | 17 | 174.36      |         |                  |
| Pure Error       | 0.000       | 5  | 0.000       |         |                  |
| Cor Total        | 8283.59     | 31 |             |         |                  |

#### 3.3.2 Modelling for PS-Fe composites

The software also predicted a quadratic model as the best fit of the experimental data in this case. The final model equation in terms of actual factors is presented in equation 4:

$$\text{Hardness (Fe)} = -3.72 + 3.08A + 0.163B + 0.099C + 0.0042AB - 0.0138AC + 0.0006BC - 0.238A^2 - 0.0021B^2 + 0.0007C^2$$

(4)

Where A is Thickness in mm, B is filler content in percentage and C is particle size in μm. The ANOVA table (Table 4) shows that the model is not significant. This means that the model is not suitable for making estimations of hardness based on these factors.

### Table 4. ANOVA Table for PS-Fe Response surface model

| Source           | Sum Squares | Df | Mean Square | F Value | p-value Prob > F |
|------------------|-------------|----|-------------|---------|------------------|
| Model            | 129.11      | 9  | 14.35       | 1.42    | 0.2399 not significant |
| A-Thickness      | 8.889E-003  | 1  | 8.889E-003  | 8.789E-004 | 0.9766           |
| B-Filler Content | 36.12       | 1  | 36.12       | 3.57    | 0.0720           |
| C-Particle size  | 29.39       | 1  | 29.39       | 2.91    | 0.1023           |
| AB               | 0.30        | 1  | 0.30        | 0.030   | 0.8646           |
| AC               | 22.96       | 1  | 22.96       | 2.27    | 0.1461           |
| BC               | 1.69        | 1  | 1.69        | 0.17    | 0.6869           |
| A²               | 15.94       | 1  | 15.94       | 1.58    | 0.2226           |
| B²               | 1.62        | 1  | 1.62        | 0.16    | 0.6929           |
| C²               | 8.03        | 1  | 8.03        | 0.79    | 0.3827           |
| Residual         | 222.49      | 22 | 10.11       |         |                  |
| Lack of Fit      | 222.49      | 17 | 13.09       |         |                  |
| Pure Error       | 0.000       | 5  | 0.000       |         |                  |
| Cor Total        | 351.61      | 31 |             |         |                  |
3.4 SEM analysis

Scanning electron microscopy (SEM) was used to obtain images of the prepared samples and analysed to determine the extent of homogeneity between the polymer resins and the metal particles. A control sample of pure polystyrene polymer was also included so that appropriate comparisons can be made.

3.4.1 Scanning of Polystyrene based resin (PBR) control sample

Polystyrene based resin (PBR) polymer prepared through solvolysis was scanned at various resolution to assess the morphology as a control sample before the injection of metallic fillers of various % composition. As shown in the Figures 10 (a-d) a visibly clear and transparent polymeric product with some fine strata behind randomly scattered white particles on the morphological surface of the sample.

![SEM images of control sample](image)

Figure 10 (a-d). SEM images showing an overall morphology of the (Polystyrene polymer-PBR) control sample

3.4.2 Polystyrene-Aluminium (PS-Al) composites at high resolution.

Figure 11 (a-b) is a SEM analysis of PS-Al composite which showed a clearly homogenously aligned smooth surfaces with few noticeable strata and agglomeration of white cloudy particles concentrated at a molecular section on the morphological surface of the sample. This denotes a stronger inter-particulate bonding and a high tendency to exhibit higher flexural, tensile and hardness.
3.4.3 Polystyrene-Iron (PS-Fe) composites at various resolutions.

Figure 12 (a-b): SEM analysis of PS-Fe composite displayed a visibly rugged and cracked material surface with white indentations. This cracked surfaces depicts poor interfacial interaction between the metal particles and the PS resin matrix. This weak cohesion between filler and resin generally weakens the composite and have been shown to negatively impact virtually all mechanical properties of the composite from moderate to low flexural, tensile and hardness strength.
Review of the SEM analysis clearly showed varied affinities in the micro-structures of the two metallic composites (PS-Al and PS-Fe.) While PS-Al metallic composite exhibited a well aligned and homogenous micro-structure with high interfacial interaction between the metal particles (Aluminium) and the PS resin matrix. This strong cohesion between filler and resin matrix resulted in a metallic composite with strong mechanical properties (high flexural, tensile strength and hardness). This is corroborated by the hardness test with an average Vickers reading of 72.2. The PS-Fe metallic composite under micro-structural analysis displayed relatively moderate to low tensile strength and hardness with weak interfacial interaction between the Iron particles and the PS resin.

4. Conclusion
The effect of composite thickness, filler content and particle sizes on the hardness of polystyrene-aluminium (PS-Al) and polystyrene-iron (PS-Fe) composites were examined. For both metal composites, lower particle size gives harder composites. A greater proportion of metal in the composite (increasing filler content) led to harder composites. Composite thickness did not have a significant effect on the hardness of the metal composites. PS-Al composite is harder than PS-Fe composite with the optimal hardness being 43.7 and 11 respectively. The factor levels that will optimise hardness for PS-Al composite are thickness of 7.5 mm, filler of 39% and particle size of 20 µm. For PS-Fe composites they are thickness of 5.9 mm, filler of 45% and particle size of 34 µm. The models obtained and the associated ANOVA revealed that particle size is the most important factor that affects the hardness of metallic plastic composites.

The two metallic composites (PS-Al and PS-Fe) exhibited contrasting mechanical strength when viewed under SEM. There was a homogenous micro-structure with high interfacial interaction between the metal particles (Aluminium) and the PS resin matrix while the PS-Fe metallic composite under micro-structural analysis displayed relatively moderate to low tensile strength and hardness with weak interfacial interaction between the Iron particles and the PS resin.

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