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Evaluation of the effects of soda-lime-silica glass with rice husk ash as an additive on the hardness behavior

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A B S T R A C T

Demand for eco-friendly materials increases each year due to their excellent properties, which has proved to contribute to developing a sustainable environment. One of the promising raw materials in producing Glass is rice husk, a waste product from paddy harvesting, containing about 90% of silica. Rice husks are usually burnt in an open area and contribute to severe air pollution problems. In this research, Soda-Lime-Silica Rice Husk Ash (SLRHA) glass which is a new combination of soda-lime silicate (SLS) glass and rice husk ash (RHA), was developed for building glass and window application. The hardness properties of the developed SLS-RHA glass system are presented in this paper. These glasses were investigated to determine the effect of RHA addition on the physical properties of SLS glass. The experimental works using RSM have successfully identified the significant factors and optimized the responses. Based on the Rockwell hardness test, the outcomes demonstrated that the glass sample contained 29.84% weight SLS and 0.06% weight RHA. The result indicated that crack propagation was increased with the increasing addition of RHA, which causes an increase in cracks and voids due to the creation of more debonding.

1. Introduction

Waste management is one of the major topics of study that is vastly developing in the scientific fields. The global markets are currently working on minimizing the necessity for extracting raw materials and maximizing the material life by promoting the reuse and recycling program (Delgoshaei et al., 2016). This idea is driven by the need to turn waste into a valuable resource by designing products that can be easily recovered and reused as raw material for a similar industry (Borek et al., 2020). Activities like these are compelling methods to avoid pollution and reduce waste emissions (Dales, 2002; Delgoshaei, 2020). Glass recycling offers a series of advantages such as the preservation of raw materials sources and helps reduce energy consumption. The soda-lime silicate (SLS) glass waste usage has been widely initiated due to the awareness in environmentally friendly products and the unique behavior of the SLS glass itself. SLS glass can be recycled several times without losing its strength and adaptability in different techniques and shapes of fabrication (Shamsudin et al., 2016). The usage of recycled SLS glass in the fabrication of glass-ceramic or green glass-ceramics composite has attracted numerous studies due to its quartz phase content (Juoi et al., 2013). Glass industries are investing significant resources in intensive research and development programs to develop new ways to use Glass, to make available new products, to enhance recyclability and effective recycling, and also to improve the energy efficiency of manufacturing sites and therefore further improve the environmental performance of glass products throughout their life cycles (Delgoshaei & Ali, 2019; Naqvi et al., 2018). Glass making requires raw materials. The most important raw materials used in glass-making are sand and recycled Glass (Burkowicz et al., 2020). The recycling of Glass is already a reality in the glass industries, although there are variances

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in how glass sectors use recycled materials (Best, 2009). Besides, waste management can help improve the sustainability of manufacturing systems (Delgoshaei & Naserbakht, 2019). Exchange of best practices and experiences on recycling is therefore key for glass industries to further increase the recycling rates of products.

In the past few years, numerous researchers have shown interest in applying rice husk (RH) silica in several fields (Pode, 2016). Due to its low thermal conductivity (κ), Rice Husk Ash (RHA) is used as an ingredient in insulation refractories. RHA has also been used widely in several fields to manufacture various silicates, zeolites, catalysts, nanocomposites, cement, lightweight building materials, insulators, and ad-sorbents over the past two decades (Hossain et al., 2018). The most common uses of RHA are in the production and manufacture of concrete. The RHA was used in the construction industry because minerals from RHA can be an admixture with high strength and high-performance concrete. Crystalline of RHA is becoming the most wanted materials in the production of ceramics, steel industries, and refractory bricks due to refractory properties in the RHA (Alazemi et al., 2021).

For countless reasons, present-day Glass is one of the most commonly used materials in daily human life, and the number is rising tremendously. Glass is known as a non-crystalline supercooled liquid and the major element in glass-making which acted as a "network-former" in glass networks, is silica (SiO_2) (Wahab et al., 2020). Huge silica resources are therefore required for glass industries. For this reason, researchers are trying to find alternative sources of silica (Mor et al., 2017). RHA has tremendous potential to fulfill this requirement because it contains more than 90 wt. % of amorphous silica (Chandrasekhar et al., 2003). For years, traditional Glass was the only material used in windows, but as polycarbonate sheeting and acrylic sheeting have gained attention, they are now both popular alternatives for window applications. Acrylic or Plexiglass windows are increasingly popular among homeowners looking for an effective and easy-to-install option instead of glass. Plexiglass sheeting is an economically conservative alternative to using Glass. Acrylic plastic sheets have 17 times the impact strength of Glass, meaning it takes a lot more force to shatter acrylic than Glass (YATAĞAN, 2019).

This work incorporates RHA into the glass matrix to optimize the response RHA to SLS Glass ratio using response surface methodology (RSM). This study aims to determine the significance of each factor and their relationship between the factors and response under the condition of the hardness test.

The idea of utilizing the RHA came out since rice husk is considered an agro-waste and has been posed as an environmental threat due to its abundance. RH is commonly burnt in the open air, which causes an enormous CO_2 emission to the atmosphere (Kartini, 2011). RH mainly contains about 75-90% of organic materials. One of the main advantages of RHA is its high content of silica, which has the potential to be the replacement of quartz sand in glass-making. SLS glass is obtained from urban waste (used glass bottles and household glass containers). Through recycling SLS waste glass, the raw material consumption is reduced, yielding economic and environmental benefits. This approach is a low investment method. Today's commercial marketplace requires innovative design elements such as plexiglass, which perfectly abide by the high quality and durability standards. Thus, using a comparison test on the hardness performance of the newly formulated SLS-RHA glass with plexiglass can determine the perceived weakness and strengths of the SLS-RHA glass. This will also help identify the important features of the product that need to be checked before its commercial release to the market.

2. Materials and Methods

2.1. Raw Material

The Bernas rice milling factory-supplied RH in Tanjung Karang, Selangor, Malaysia and contains almost 75 to 90% of organic materials such as cellulose, lignin, and hemicellulose (Beidaghy Dizaji et al., 2019). RH was pre-treated with distilled water and leached with 1 M HCL solution at standard room temperature to remove the other impurities material and dirt in the rice husk. The optimum ratio for HCL was 100 mL and was poured into a beaker with an amount of 10 grams RH. A standard Magnetic stirrer in UPM Mechanical Engineering Lab was used to heat the beaker. Once the temperature reached 60 °C, a vigorous stirring of the mixture started for 30 minutes continuously along the process. The thermometer was constantly put into the beaker to ensure the 60 °C temperature was throughout the mixture. Unwanted HCL was required to be discarded carefully as it could be corrosive to others. The RHA was filtered using a standard stainless-steel mesh screen and washed completely with distilled water to remove any remaining HC solution on and solid residue.

RH was then dried in an oven for 2 hours at 110 °C. This process was to ensure the RH was completely dried before the incineration step. WiseTherm Digital Muffle Furnace (Daihan, Gangwon, South Korea) was used to incinerate dried RH into RHA with a controlled temperature of 600 °C for two hours. The RHA produced are then ground into fine particles size using pulverizing machine RT-02A (Mill Powder Tech, Tainan, Taiwan) and sieved using Endecotts Laboratory Test Sieve (Endecotts, London, United Kingdom) to obtain an average particle size below 75 μm.

2.2. Preparation of SLS-RHA Glass

The materials needed for fabricating the Glass are soda-lime silicate (SLS) glass powder and rice husk ash (RHA). To produce SLS glass powder, the waste SLS glass bottle was crushed using mortar and pestle until the glass powder was ground to the size of smaller than 200 μm. The SLS glass powder is then mixed with differratiosatio of RHA. Samples of RHA glass with three duplications were prepared using a melt quenching technique. The ratio of the RHA is 0%, 5%, and 10% from the total mass sample. The total mass of the mixture is 30.0 g that will be placed into the glass mold after the heating process. The total sample powder weight for each series is 30.0 g.

SLS powder and RHA were mixed using a dry milling machine to obtain a homogenous sample powder. The milling process was continued for 2 hours and repeated for another sample with different compositions. After the milling process, the sample powder was transferred to an alumina crucible. To reduce the tendency to volatilization, the crucible containing the sample mixture powder was required to be preheated at 450°C for a period of one hour. After the preheating process, the crucible was transferred into the electric furnace for the melting process at 1400°C. The fabricated SLS-RHA glass is shown in Fig. 1 and the process flowchart for glass fabrication is shown in Fig. 2.

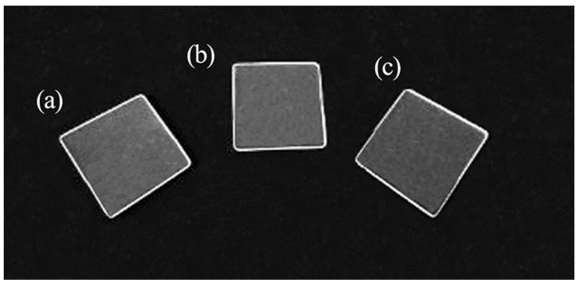

Fig. 1. SLS-RHA glass samples (a) S1 (b) S2 and (c) S3

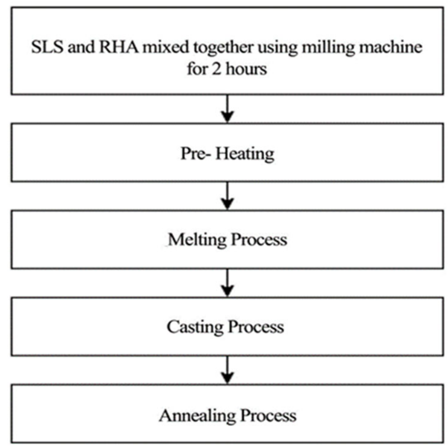

Fig. 2. Glass fabrication process

2.3. Preparation of SLS-RHA Glass

Response surface methodology (RSM) has developed a model for predicting the Rockwell hardness value (RHV). Design Expert 11.0 was used for the RSM analysis. The central composite design (CCD) method was used for the model design. The CCD involves 13 experimental runs for the variables SLS glass (A) and the percentage of RHA (B) in this experimental design.

2.4. Rockwell Hardness Test

FUTURE-TECH Rockwell Hardness Tester FR Series, as shown in Figure 3 was used in this study to test the hardness of glasses. Rockwell hardness test has the scaling based on the application and the materials. In this research, the HRF scale was used to determine the hardness of SLS-RHA glass. A 1/16-inch ball was selected as an indenter in this research project with a 60 kg load.

Fig. 3. FUTURE-TECH Rockwell Hardness Tester FR Series

2.5. Microstructure Analysis and Material Characterisation

SEM was used to analyze external morphology such as texture, chemical composition, crystalline structure, and the orientation of the materials of the samples. The microstructure was outlined by exposing the surface structure of material underneath a microscope with at least 25× magnification. SEM imaging was operated using a Hitachi S-3400 N (Hitachi, Tokyo, Japan) for the samples' particle study and compound representation. It was conducted at 15 kV. The importance of the SEM result is used to prove and support the information about the structure of SLS-RHA glass before and after the Rockwell hardness test.

X-Ray Fluorescence (XRF) was conducted using Olympus Handheld XRF Analyzer - Delta Professional to determine substance composition in the SLS-RHA glass. Powdered samples of SLS-RHA glass were used in this test. XRF is a non-destructive diagnostic method to reveal the percentage of every primary component of a substance. Fundamentally, it works by evaluating the X-ray released from the sample after the primary X-ray source energized onto the sample.

3. Results

The complete design matrix and HRF responses values are given in Table 1.

Table 1. Experimental Design for Rockwell Hardness Test HRF Value

Sample	SLS (wt. %)	RHA (wt. %)	HRF
S1	27.00	3.00	101.4
S2	28.50	1.50	119.5
S3	29.00	1.00	124.6
S4	30.00	0.00	128.9
S5	28.50	1.50	117.4
S6	29.00	1.00	124.5
S7	28.50	1.50	118.4
S8	27.00	3.00	102.4
S9	29.00	1.00	126.6
S10	28.50	1.50	118.1
S11	28.50	1.50	120.9
S12	27.00	1.50	119.1
S13	28.50	1.50	118.2

3.1. Statistical analysis of hardness properties

The summary for Analysis of Variance (ANOVA) of the developed RSM model for RHV HRF is given in Table 2. It can be seen that the established response model was significant on 97.30% of confidence level. The Model F-value of 107.98 implies the model is significant with only a 0.01% chance that a "Model F-value" this large could occur due to noise (Delgoshaei & Ali, 2017). The values of p less than 0.0500 indicate model terms are significant. In this case B, AB are significant model terms. Values greater than 0.1000 indicate that the model terms are not significant.

Table 2. Results of analysis of variance (ANOVA) for HRF response model

Source	Sum of Squares	Degree of Freedom	Mean Square	F-Value	*p*-Value
Model	785.35	5	261.78	107.98	< 0.0001
A	0.0913	1	0.091	0.038	< 0.8505
B	123.69	1	123.69	51.02	0.0001
AB	24.88	1	24.88	10.26	0.0108
Residual	21.82	9	2.42	-	-
Cor Total	807.17	12	-	-	-
Model	785.35	5	261.78	107.98	< 0.0001

Table 3 indicates a model that can navigate the design space. The developed response model in terms of the coded and fundamental factors with all the model terms can be used to predict the Rockwell hardness value of HRF. The regression models can be used to calculate and analyze the effect of factors on Rockwell Hardness performance of SLS-RHA glass. The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are constant. The intercept in an orthogonal design is all the runs' overall average response. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal, the Variance Inflation Factors (VIF) are 1; VIFs greater than 1 indicate multi-collinearity. The higher the VIF, the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

Table 3. Model to navigate the design space

Factor	Coefficient Estimate	Degree of Freedom	Standard Error	95% CI Low	95% CI High	VIF
Intercept	119.42	1	0.55	118.18	120.66	-
A-SLS	0.32	1	1.65	-3.41	4.06	4.79
B-RHA	-13.81	1	1.93	-18.18	-9.43	5.14
AB	3.64	1	1.14	1.07	6.22	1.16

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative factors by comparing the factor coefficients. Final Equation in Terms of Coded Factors:

$$YHRF = 119.42 + 0.32\,(A) - 13.81\,(B) + 3.64\,(AB). \quad (1)$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. The levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space. Final Equation in Terms of Actual Factors:

$$YHRF = 196.36 - 2.22\,(A) - 55.35\,(B) + 1.62\,(AB) \quad (2)$$

3.2. Effect of factors on hardness test

ANOVA and the regression models were used to analyze the effect of SLS glass to RHA weight percentage on the hardness properties. Contour plots were used for better illustration. Fig. 4 illustrates the effect of SLS glass (A) and RHA weight (B) on Rockwell HRF response. Observation in Fig. 4 showed that a higher percentage of A: SLS and a lower percentage of B: RHA resulted in higher HRF values.

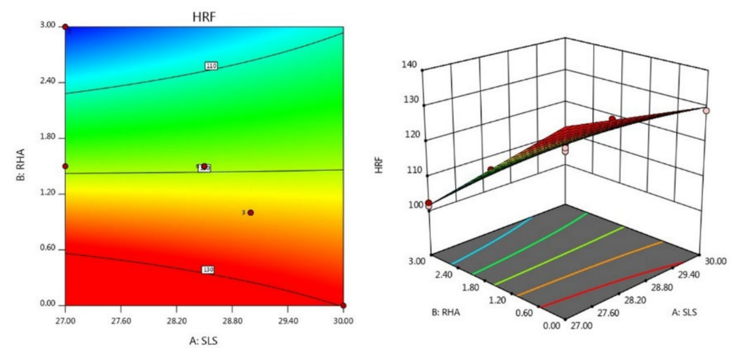

Fig. 4. Contour diagram for Rockwell hardness HRF value

3.3. SLS-RHA glass optimization

RHA is the main addition to this glass network. Thus, the main goal is the highest RHA variable setting with near-optimal properties for hardness value. The objective was to maximize the Rockwell hardness value, thus the target values that are the goal to be achieved were set at the lowest value and the upper value was set at the highest importance. The constraints to obtain numerical optimization are shown in Table 4.

Table 4. Numerical optimization constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:SLS	is in range	27	30	1	1	3
B:RHA	is in range	0	3	1	1	3
HRF	maximize	101.4	128.9	1	1	3

Fig. 5 illustrates the predicted optimum conditions and the responses studied for Rockwell HRF value. The optimization plot on the effect of the different combinations of factor settings on the Rockwell responses and the contour plot is shown in Fig. 6. The predicted optimum operating parameters influencing HRF value were estimated to be SLS (29.84% wt.) and RHA (0.06% wt.). At these optimum conditions, the corresponding predicted HRF value was 129.295. The desirability of optimization was calculated as 1.000, indicating that all parameters were within the target to obtain the maximum Rockwell responses value.

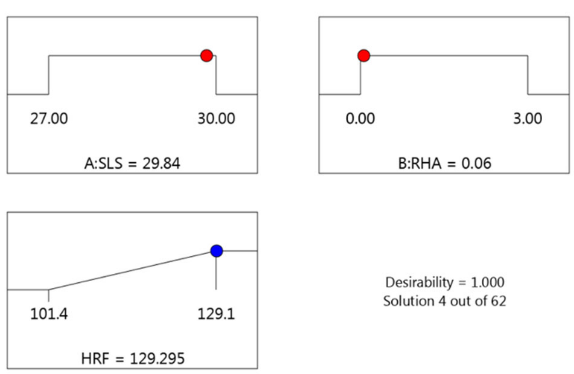

Fig. 5. Optimum conditions and response for Rockwell HRF properties

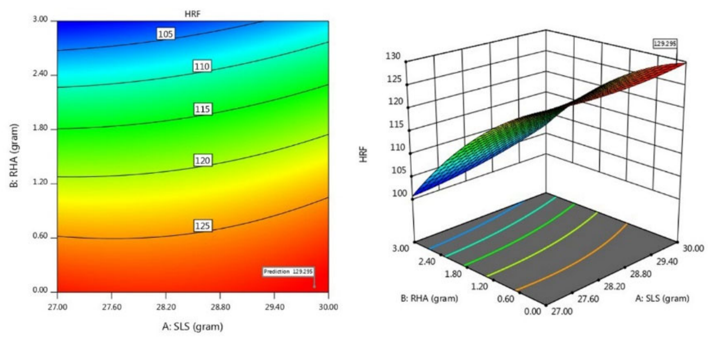

Fig. 6. Rockwell hardness HRF response optimization contour plot

3.4. Experimental Validation

Experimental validation is the final step in the modeling process, and it verifies the model's accuracy. Three validation experiments were carried out under the optimal conditions obtained from the optimization plot, as shown in Figure 5, to verify the reproducibility of the established regression model and the RSM model. For a nonlinear process, the optimization and validity of the model are only verified when the average difference between experimental and predicted values is less than 15% (Milkey et al., 2014). Table 5 shows the experimental validation for fire resistance properties; it was found that the average errors for the HRF values were well below 15% at 2.02%. It was concluded that the developed regression model established using this method could optimize the value for the responses.

Table 5. Experimental validation for the Rockwell hardness test.

	HRF		
	Experimental Value	**Predicted Value**	**Error (%)**
SV_1	127.2	129.3	1.65
SV_2	126.1	129.3	2.53
SV_3	126.9.	129.3	1.89
	Error		2.02

SV = Sample validation.

3.5. SLS-RHA Glass Material Characterization

For material characterization, design matrix and response values for samples S1 and S3 were discussed in detail as these samples had the best and worst performance in the Rockwell hardness test. Hardness refers to stiffness or temper or to resistance to bending, scratching, abrasion, or cutting (Pendke et al., 2019). It also gives the ability to resist bending permanently and resist deformation when a load is applied. The greater the hardness of the Glass, the more excellent resistance to deformation (Sawamura et al., 2018). XRF analysis for the SLS-RHA samples was performed and the results obtained from the analysis were tabulated in Table 6

Table 6. Experimental validation for the fire resistance test

Element	**Concentration (wt. %)**	
	S1	**S3**
SiO_2	61.36	67.32
K_2O	2.36	0.62
Al_2O_3	9.43	10.55
Fe_2O_3	0.85	0.01
CaO	1.43	0.74
MgO	8.45	9.79
Na_2O	11.23	9.66
P_2O_5	0.09	0.07
Others	4.79	1.23

The XRF analysis identified eight oxides with primarily SiO2, Al2O3, Na2O and MgO in the SLS-RHA glass. The highest percentage of SiO2 content was traced in sample S3 with 67.32%, while S3 with 61.36%. Slight traces of P2O5, Fe2O3, and metal oxides are also observed. It was observed that with the increase in RHA percentage, the SiO2 content also increased. The XRF composition results agree with (Andreola et al., 2013), which found that the main components in RHA glass compositions are SiO2, Al2O3, Na2O3 and MgO. The study of microstructure is significant to analyze the glass behavior when the impact is applied. The surface of the glass samples for S1 and S3 were analyzed using SEM. The SEM images at 100 μm obtained from the upper surfaces and further magnified 5 μm at the point of impact for sample S1 are shown in Figure 7. For sample S1, there is no cracking observed and the interaction between the indenter and sample S1 glass surface only resulted in a piece of Glass being chipped off from the surface of the Glass.

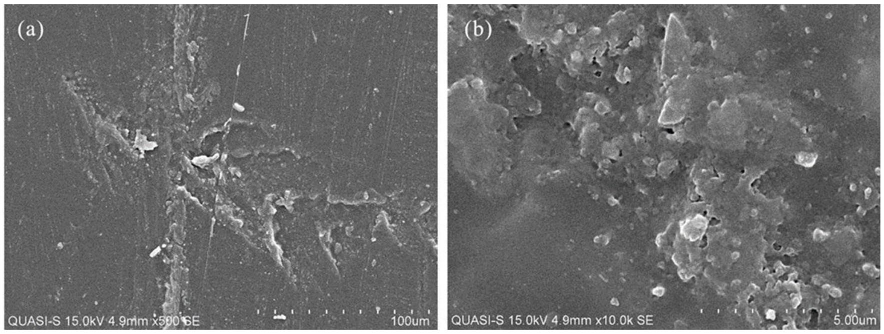

Fig. 7. Sample S1 magnified (a) 100 µm upper surfaces (b) point of impact at 5 µm.

The SEM images at 100 µm obtained from the upper surfaces and further magnified 5 µm at the point of impact for sample S3 are shown in Fig. 8. From SEM observation, sample S3 shows a distinct line of crack damage. The result indicated that crack propagation was increased with the increasing addition of RHA, which causes an increase in cracks and voids due to the creation of more debonding. These cracks are due to non-bridging oxygen (NBO) in the SLS-RHA glass. The formation of NBOs will decrease the connectivity of the glass network and the structures of the Glass become weakened (Khalil et al., 2010). The level of porosity influences all elastic properties since pores act as the second phase of a zero modulus. Based on the SEM image in Figure 9, it was found that the SLS-RHA glass for S3 is more porous and rougher, which supports the reason sample S3 has more cracks and bigger crack propagation.

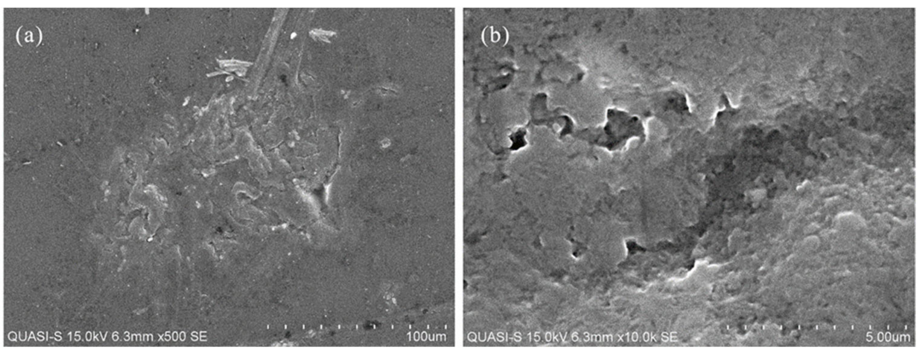

Fig. 8. Sample S3 magnified (a) 100 µm upper surfaces (b) point of impact at 5 µm

5. Conclusions

A new combination of Glass is produced using SLS waste with added RHA content. These Glasses were investigated to determine the effect of RHA addition on the physical properties of SLS glass. The experimental works using RSM have successfully identified the significant factors and optimized the responses. Experiments on the thermal proper-ties conducted were based on the design matrix generated by the Design-Expert version 11 software (Stat-Ease Inc., MN, USA) and then carried out on the laboratory scale following the appropriate standards. Based on the Rockwell hardness test, the outcomes demonstrated that the glass sample contained 29.84% weight SLS and 0.06% weight RHA. The result indicated that crack propagation was increased with the increasing addition of RHA, which causes an increase in cracks and voids due to the creation of more debonding. The formation of NBOs will decrease the connectivity of the glass network and the structures of the Glass become weakened. With the regulations about the environment and waste disposal becoming stringent day by day, the glass-making industry is compelled to adopt a zero-waste policy for sustainability. Thus, utilizing high SiO2 content in RH and recycled SLS glass is an innovative solution to transform "Waste to Wealth."

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