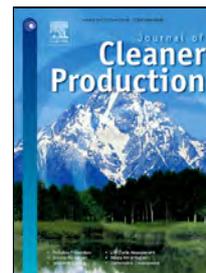


Accepted Manuscript

From waste glass to building materials – an innovative sustainable solution for waste glass



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PII: S0959-6526(18)31250-2
DOI: 10.1016/j.jclepro.2018.04.214
Reference: JCLP 12795
To appear in: *Journal of Cleaner Production*

Received Date: 29 May 2017
Revised Date: 16 April 2018
Accepted Date: 23 April 2018

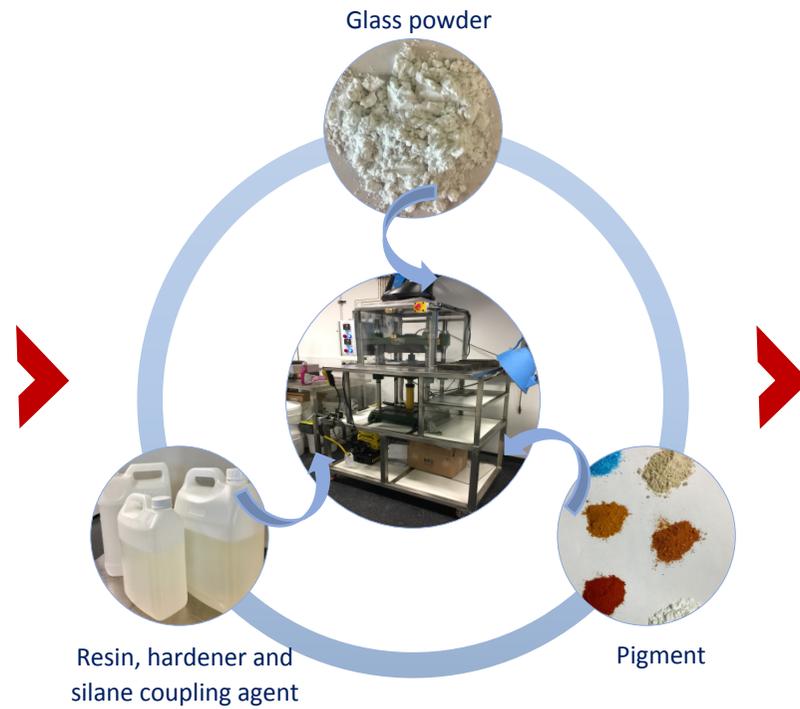
Please cite this article as: Heriyanto, Farshid Pahlevani, Veena Sahajwalla, From waste glass to building materials – an innovative sustainable solution for waste glass, *Journal of Cleaner Production* (2018), doi: 10.1016/j.jclepro.2018.04.214

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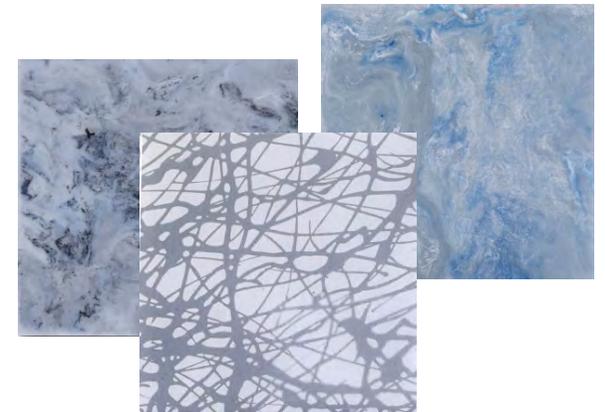
Material recovery from urban waste stream



Functional Raw Materials



Polymeric glass composite



From waste glass to building materials – an innovative sustainable solution for waste glass

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Abstract

This report details a cost-effective new process for transforming mixed waste glass into high-value building materials without remelting. Worldwide, waste glass is a growing burden and new options are urgently needed for the large volumes of speciality, mixed, broken and contaminated glass that cannot currently be recycled. Conventional glass recycling technologies are limited by the need to separate waste glass into different glass types and the extreme sensitivity of the remelting process to any contamination. In this study, we demonstrated that mixed broken glass could be used as primary input in the production of polymeric glass composites. The composites' mechanical properties, utility, aesthetic appeal and expected market value were comparable to natural and engineered stone products, widely used as kitchen and bathroom benchtops and floor and wall tiles. Waste glass powder with an average particle size smaller than 108 μm was ground then mixed with resin as a binder before the mixture was hot-pressed under pressure. Various ratios of waste glass to resin binder, as well as the effect of a coupling agent and reinforcing mesh were tested. The optimum PGC achieved a flexural strength of 48 MPa, water absorption below 0.002%, a density of 2.113 kg/m³ and compressive strength of 101 MPa with minimum scratch, wear and UV degradation behaviour. Its excellence in the mechanical value stand in parallel with engineering stone but, with cheaper and efficient process. Additionally, this new recycling process embodies an important unique alternative to the remelting of waste glass with the potential to deliver economic and environmental benefits wherever waste glass is stockpiled.

Keywords: Polymeric glass composite panel; low-carbon material; waste glasses recovery.

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1. Introduction

Over the past century, the total global consumption of materials has increased eight-fold and per capita consumption has risen from 4.6 tonnes to 10.3 tonnes a year (Krausmann et al., 2009), matched by the generation of vast volumes of waste. Recycling technologies and numerous national and local regulations intended to drive the recovery of resources from waste have failed to keep pace with this accelerating consumption, resulting in ever-increasing volumes of waste to landfill (EPA, 2017). One major waste stream emerging as a serious challenge worldwide is glass, despite its ideal properties for recycling and its unsuitability for landfilling. Although glass can repeatedly be recycled with no loss of quality, large volumes of glass are ending up in the landfill. In Australia, for example, in the five years to 2015, glass consumption increased but the national glass recycling rate fell from 49% to 42% (IndustryEdge Pty Ltd and Equilibrium OMG Pty Ltd, 2016; Adaway et al., 2015), leaving almost 800,000 tonnes of waste glass requiring disposal every year (FEVE, 2017). Glass recycling rates in European Union nations are the world's highest, averaging 73% (Glass Packing Institute, 2016), but other countries are trailing much further behind, such as the US (34%) and Singapore 20% (National Environment Agency, 2017). One major barrier to conventional recycling is the need to both separate glasses into its various types. Some speciality glasses like window panes, tempered glass, Pyrex, borosilicate glass and broken glass are usually not considered recyclable (Gautam et al., 2012). As the melting points and characteristics of different glass types are precisely matched to their uses, mixed glass cannot be remelted in one batch. If heat treated glass is mixed with bottle and jar recycling process, it can result in a different viscosity of the glass melt and prevent the molten glass to be extruded properly (Planet Ark Environmental Foundation, 2010). Forming, cooling rate and an annealing temperature of mixed glass are also harder to control. Additionally, different types of glass have different coefficient of thermal expansion which might expand and contract at a different rate. Mixing these glasses results in cracking or shattering of the piece during cooling (University of Delaware, 1843).

Besides, contaminations are also the major problem in conventional glass recycling. Demolition sites are a major source of waste window glass, but an effort to recycle old window glass cullet to new glass has not been done. Unlike container glass which allows a small amount of impurities of 20- 50 ppm, production of flat window glass tolerate no ceramic and ferrous metal impurities (Vieitez et al., 2011). Also, various types of window glass ranging from

standard, tempered, silver-based varnish (mirror) to laminated glass have a different chemical composition and impurities, preventing conventional recycling to be done. To increase glass recycling rates, alternatives means of reprocessing waste glass without remelting are needed.

Previous studies have reported the use of the waste glass powder (GP) as a filler for asphalt, concrete pavements and sidewalk slabs and as an alternative to both fine ([Glass packing Institute, 2016; Hyperphysics, 2014](#)) and coarse aggregates ([Kenneth et al., 2013](#)) in concrete. However, as waste glass can be used to replace only 10-30% of the original materials in these applications, they can absorb only modest volumes of this bulk waste ([Glass packing Institute, 2016; Hyperphysics, 2014](#)).

It has also long been known that fine silica powder can be used to improve the mechanical properties of cement. The fine silica powder reacts with the alkalis in cement to form more calcium silicate hydroxide compound (CSH) - the primary binder in cement ([Liang et al., 2007](#)). As glass contains around 72% silica and sand contains 89-96% silica ([Manger, 1934](#)), glass waste has been investigated as a potentially promising partial substitute for sand in concrete. However, while glass powder in cement is helpful in the production of CSHs its sodium carbonate content releases more alkalis into the concrete matrix when it is used in place of a proportion of sand ([Glass packing Institute, 2016; Hyperphysics, 2014](#)). This further enhances alkali-silica reactivity (ASR), a reaction that may cause expansion and cracks in concrete over the long term ([Liang et al., 2007](#)). This means that substituting sand with glass waste in concrete results in 'down-cycling' and is of little economic advantage. And, as these processes can absorb only moderate volumes of waste glass powder without remelting, their impact is limited.

This report aims to develop a cost-effective new process that transforms large quantity of mixed waste glass into valuable polymeric glass composite (PGC) panels similar in look and performance to various natural and engineered stone products currently on the market. The production process is quite similar to that used in engineered stone, as described by other researcher ([Lee et al., 2008](#)) but without the necessity of advanced machinery. Three main materials i.e. waste glass, resin, and silane coupling agent will be used and their role will be described thoroughly in this study. Additionally, the scope will include an investigation of resin to glass powder ratio along with the optimum processing condition to create defect-free products. High pressure and exact ratio of binder and powder are found to be the key constraint. Vacuum vibrator compaction, used to remove air bubble in engineered stone, are therefore not essential. Also, for a theoretical purpose, this paper will cover the effect of coupling agent in

modifying the structure of glass powder surface and improving its wettability as well as adhesion with the resin binder. SEM analysis of the fracture surface of the non-treated and treated samples will also be conducted and their difference in mechanical strength will be reported and compared with natural and engineered stones in the market.

Unlike engineered Quartz stone in general, PGCs contains more than 80% waste glass which represents an environmentally and economically desirable new pathway for waste minimization. Additionally, the aggregates, i.e. marble, granite and quartz fragments which are the main components in engineering stone are omitted and are substituted with a large proportion of waste glass powder. The advantage of using glass over some natural stones lies in its general characteristics: glass has an intermediate Mohs hardness of 5.5-6, is non-porous, amorphous, highly water resistant and moderately resistant to fire and high temperatures (Prat et al., 2016). The design of the polymeric glass composite panels replicates the natural look of marble or granite while delivering better flexural and compressive strength, as well as low porosity and water absorption. For practical purpose, the PGC panels are designed to serve as cost-effective, high-quality benchtops for kitchens and bathroom vanities and as wall and floor tiles. The mechanical properties of the glass composite panels were tested based on the American Society for Testing and Materials (ASTM) standard for similar dimension stone.

2. Materials and methods

2.1. Raw materials characterisation

2.1.1. Waste glass powder

For this study, waste window glass, tempered glass, laminated glass and borosilicate glass were mixed to replicate the diverse glass waste stream. The chemical composition of the various glasses was analysed by using X-Ray Fluorescence (XRF), as shown in Table 1. All the glass types, except borosilicate glass, contained mostly SiO_2 , Na_2O , CaO , with a small proportion of Al_2O_3 and MgO . Borosilicate glass has a slightly higher percentage of SiO_2 and contains B_2O_3 rather than CaO . Unlike Quartz powder which was made from crystalline silica, the SiO_2 in the waste glass is amorphous as shown by X-ray diffraction (XRD) analysis. Although amorphous SiO_2 does not offer extraordinary properties as crystalline SiO_2 in Quartz, amorphous SiO_2 retains its general characteristics of low thermal expansion, high melting

point, medium hardness and good abrasion resistance. It deserves consideration as raw materials replacement of Quartz powder in countertop production.

Scanning Electron Microscope (SEM) analysis in Fig. 1 also showed that glass powder has compact irregular oblong shape particles. These angular surface topography lead to an increase in cohesion (the ability of the glass powder to stick together) and internal friction angle (grain-grain friction resistant) (Rong et al., 2013). These factors might decrease workability as the glass powder tend to clump together (Penn State Engineering, 1855). Vigorous mixing is therefore needed. On the contrary, the advantages of having filler with high friction angles are that they induce high shear yielding in the final product which results in higher strength. The glass powder could, therefore, be valuable filler in countertop slab production.

Table 1

XRF elemental analysis of different types of glasses in weight percentage (wt%).

Waste glasses	SiO ₂	Al ₂ O ₃	MgO	CaO	Na ₂ O	Fe ₂ O ₃	B ₂ O ₃	Others
Window (float glass)	71.216 %	1.087%	3.628%	8.931%	14.387%	0.174%	0.000%	0.577%
Laminated glass	71.596 %	0.051%	4.090%	9.273%	13.955%	0.082%	0.000%	0.953%
Borosilicate glass	75.626%	2.258%	0.026%	0.013%	4.590%	0.006%	15.640%	1.841%
Tempered glass	72.187 %	0.067%	4.095%	9.377%	13.875%	0.116%	0.000%	0.283%
Mixed glass	72.656%	0.8658%	2.959%	6.899%	11.702%	0.0945%	3.910%	0.9135%

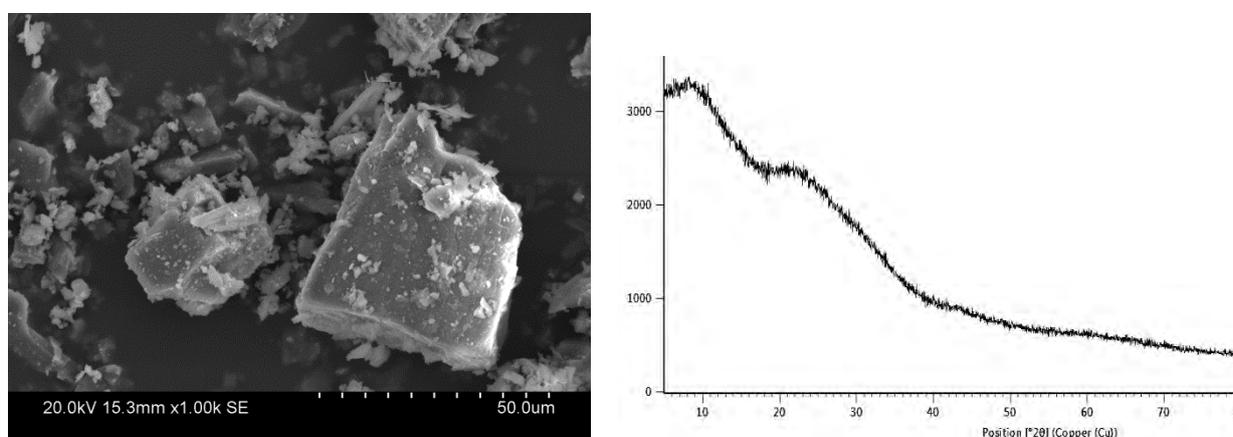


Fig. 1. (A) SEM and (B) X-ray diffraction analysis of glass powder

2.1.2. Binder

The resin used in this study was modified epoxy casting resin with characteristics of medium viscosity, non-toxic, good chemical and abrasion resistance and high UV resistant. The resin was mixed with hardener with a volume ratio of 2 to 1. The resin became gelled within 20-40 minutes under isothermal reaction at room temperature. During this process, the viscosity of the liquid resin increased with curing time to form a clear solid block. The resin used in this study is used explicitly for countertop slab production and has significant resistance to UV degradation. Fig. 2 represents the yellowing effect of the corresponding products in comparison to general resin when laid under direct sunlight for 42 days. The modified resin only showed minor colouration with its 42 days-yellowing rating being equivalent to that of 7 days-yellowing rating in general epoxy. The result proved that the modified resin had significantly higher resistant to UV degradation. Similar to engineering stone sold commercially, irrespective of the high UV stability of the resin used, the PGC produced is recommended to only be used indoor.

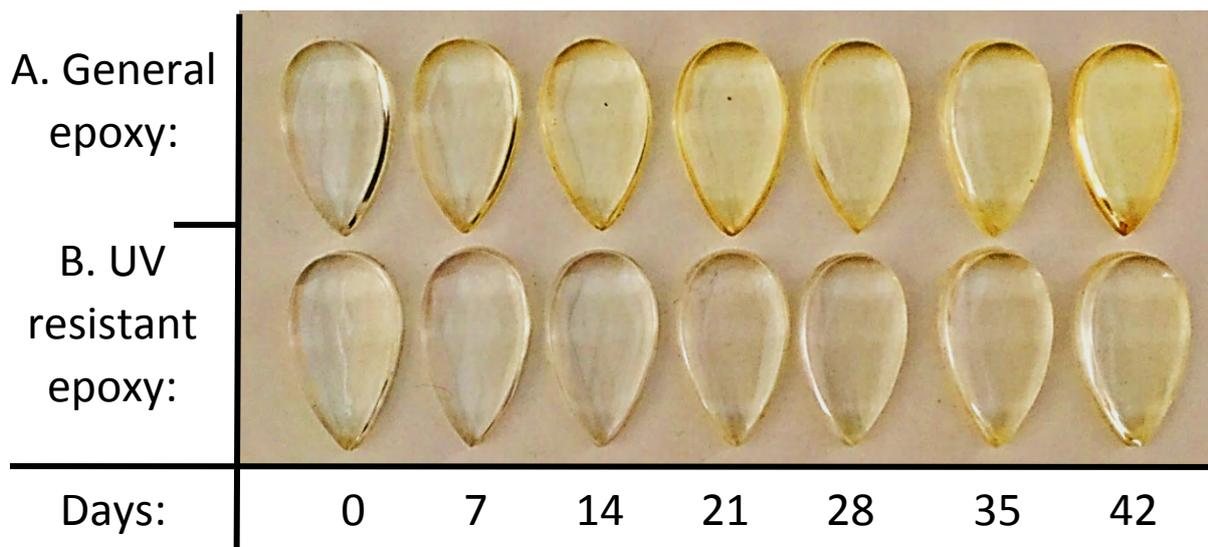


Fig. 2. Yellowing effect of (A) General epoxy and (B) UV resistant epoxy

2.1.3. Coupling agent

In a composite system, interactions between organic and inorganic materials tend to offer an inferior bonding adhesion capability due to the poor wettability on the surface of these two components. Resin binder contains hydrocarbon which is non-polar (hydrophobic), whereas glass powder is polar (hydrophilic). Therefore, obtaining good adhesion is relatively difficult, resulting in low tensile and flexural strength.

The interfacial adhesion in composite panels can, however, be improved by surface modification with the introduction of a coupling agent. Silane coupling agents are typically used for glass- polymer resin composites with one of the reactive groups binding with the surface of the inorganic materials and the other being copolymerised within the polymer matrix. The silane coupling agent used in this study is β - (3,4 epoxy cyclohexyl)- ethytrimethoxysilane (CAS no. 3388-04-3) from Guangzhou Double Peach Fine Chemical Co., Ltd. The schematic of the interfacial modification is shown in Fig. 3 where Y is an organic base group with $-(\text{OCH}_3)_3$ reacted with water to form a reactive silanol (Si-OH). The diluted coupling agent ($\text{Y-Si}(\text{OH})_3$) was mixed with inorganic glass powder surface to form a slurry. It was then dried in an oven at 100°C overnight, leaving only silane-treated glass powder. From these reactions, the bridge between the organic base group of coupling agent and glass surfaces was built and the surface properties of the glass powder were improved to establish a bonding capacity with resin.

Reaction 1:



Reaction 2:

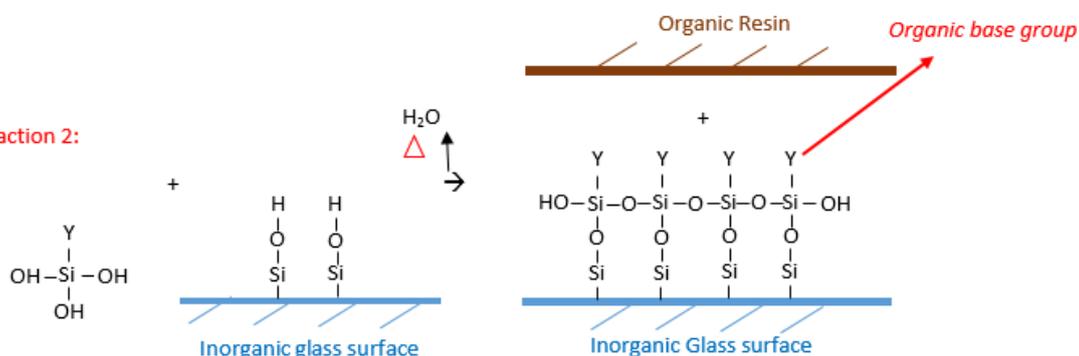


Fig. 3. Interface modification of glass powder and resin with the optimum amount of silane coupling agent

2.1.4. Fibreglass sheet

A sheet of fibreglass mesh can be added as a reinforcement to improve the flexural strength of the composite panels where required. While the sheet is not essential, it is recommended for thinner slabs, with narrow widths, which are made for table or countertop applications. In this study, the fibre glass was added in the tension region, as shown in the Fig. 4A, as this where cracks start to propagate. While the fibre mesh works effectively to take the tensile load in these regions, it will provide no bending improvement if is placed in the middle or compression region (Massarelli, 2000).

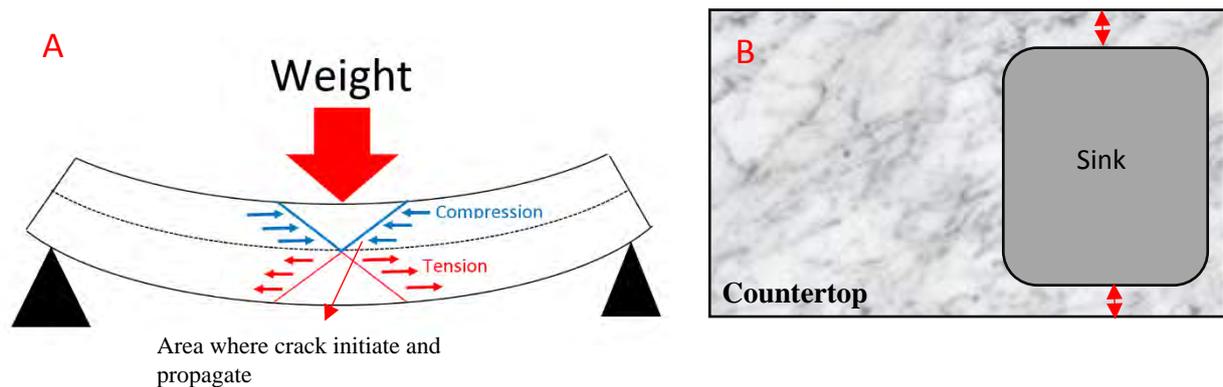


Fig. 4. (A) Compression and tension region under compression load (National Ready Mixed Concrete Association, 2003). (B) Thin narrow area (in red) suitable for fibre mesh reinforcement

2.1.5. Pigment

To create different appearances and designs, synthetic dye or coloured waste powder from ochre stone, hematite, and carbon was added. Copper and aluminium powder from e-waste could also be a useful addition to create glitter effects in the polymeric glass composite slabs produced.

2.2. Manufacturing process and formulation

Fig. 5 illustrates the material preparation method and production steps taken to produce the polymeric glass composite panels. The raw materials were subjected to eight steps process. The process consisted of crushing, grinding, pre-treatment of the glass powder, drying, mixing, moulding, hot pressing and cooling for disassembly. First, the mixed waste glass collected was crushed using a hammer or jaw crusher into 3-4 cm size aggregates and dried in an oven for 24 hours at 60°C to remove any moisture. The waste glass cullet was then ground into fine powder using ring mills. Inside this machine, the sample was ground through vibration motion mechanism and was only suitable for brittle materials. During this process, if laminated glasses were introduced, the PVB layer would stay in 1 or 2 cm diameter globe and were easily removed by sieving through a 108 µm metal screen. At this stage, the glass powder was termed 1 (GP1) in the schematic. When a silane coupling agent was used, further treatment was needed. Consequently, the glass powder 1(GP1) was then dispersed in the solution of diluted alcohol and silane coupling agent to form a slurry. The alcohol from the slurry was evaporated in an

oven overnight. After drying, the slurry formed a chunk of compacted powder. The compacted powder was then again ground using ring mill to obtain glass powder 2 (GP2).

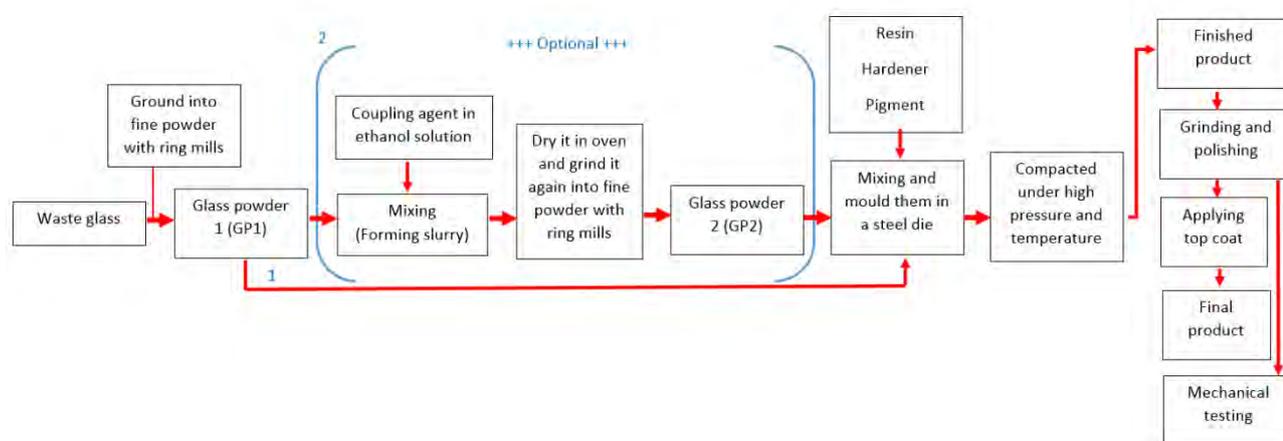


Fig. 5. Schematic of experimental procedure

The waste glass powder (GP 1 or 2), resin, hardener and 0.5-2% pigment was combined in various proportions, as per formulae in [Table 2](#), and mixed vigorously for at least 5 minutes to ensure complete mixing and homogeneity. Following the procedures, the blend was hand-laid in a 240x 240 mm carbon steel die, lined with a non-stick Teflon sheet. The mixture was flattened and sealed with a square steel lid. The sealed die was loaded into a hydraulic hot press which has been pre-heated at 80°C. It was then compacted under pressure of 550 bars for 30 minutes. The mould was then cooled to room temperature for at least 1/2 hours before the sample was removed from the steel mould.

Table 2

Panels formulation and design parameters in weight percentage (wt%).

No.	Panel type (GP: Resin compound)	Glass powder ($<108 \mu\text{m}$) in weight percent	Resin compound		Silane coupling agent in percent (relative to glass powder)	Pigment
			Part A resin in weight percent	Hardener (Part B) in weight percent		
1	A (65:35)	65	23.333	11.336	-	Pigment was only added for aesthetic purpose. The
2	B (70:30)	70	20	10	-	
3	C (75:25)	75	16.667	8.333	-	
4	D (80:20)	80	13.333	6.667	-	

5	E (85: 15)	25	10	5	-	percentage varies depending on the targeted colour in the final product. Maximum pigment added is 2% to prevent any effect on the mechanical properties.	
6	F (80:20) + 1% silane (silane percentage relative to glass powder)	80	13.333	6.667	1		
7	G (80:20) + 2% silane	80	13.333	6.667	2		
8	H (80:20) + 3% silane	80	13.333	6.667	3		
9	I (80:20) + 4% silane	80	13.333	6.667	4		
10	J (80:20) + 5% silane	80	13.333	6.667	5		
Note: Powder glass filler along with resin binder account for 100% wt. Coupling agent was added relative to powder filler and is added after everything else is measured.							
11	Marble stone	Reference samples available in the market.					
12	Granite stone	These samples were cut and their mechanical properties were measured and compared with the PGCs produced.					
13	Quartz stone						
14	Engineering stone						

2.3. Mechanical testing procedures

The composite panels were further cut and polished into required slabs with sharp edges removed for mechanical testing. The panels were tested based on American Society for Testing and Materials (ASTM) standard and were designed for countertop use. The test includes bending, compression, wear and scratch resistant, water absorption and thermal degradation test. At least 5 specimens were prepared for each test with the average value reported in the result. Unlike ceramics, the percent error of the specimens tested was relatively low with a standard deviation of less than 5% due to the homogeneity in the produced samples and ductile properties retained from the resin binder.

2.3.1. Four-point bending test

The flexural strength or modulus of rupture (MOR) of material is defined as its ability to resist deformation under load. This property is especially important when assessing the

performance of engineered stone, or comparable products. The flexural strength value in this study was measured based on International standard ASTM C880/880M using Instron 5982 universal mechanical testing machine. Load at a uniform stress rate of 4 MPa/min was applied to failure. The dimension of the specimen tested was 240 x 100 x 18 mm with span of 180 mm.

2.3.2. Compression test

The compressive test is used to measure the maximum amount of compressive load of a material can bear before fracture. The compression value in this study was measured based on International standard ASTM C170/C170-16 using Instron 5982 universal mechanical testing machine. At least 8 specimens were tested in perpendicular and parallel orientation. However, no significant difference was found in both orientations. The dimension of the specimen was 18 x 18 x 18 mm³ with a ratio of the height and diameter in error range of 0.9:1.0 and 1.1:1.0). Load at a uniform rate of 0.5 MPa/s was applied until the specimen failed.

2.3.3. Water absorption

It is essential to measure the water absorption behaviour to determine the durability of the PGCs when exposed to high moisture environmental conditions. The samples were first weighed dry, then immersed in water for 24 hours. They were then surface dried with a damp cloth and weighed to the nearest 0.01 gram. By measuring the weight difference between the dry and wet samples, water absorption can be calculated based on the equation 1.

$$\text{Absorption, weight\%} = \left[\frac{(B - A)}{A} \right] \times 100 \quad (1)$$

Where

A = weight of the dried specimen, (g) and

B = weight of the specimen after immersion, (g)

2.3.4. Thermogravimetric analysis

The thermogravimetric analysis (TGA) was measured by PerkinElmer STA 6000 in an inert nitrogen atmosphere with a flow rate of 20 l/min. The analysis measured mass of a sample over time as temperature changes. In this study, the TGA was used to identify the minimum temperature when the sample degraded (thermal degradation) which was also the maximum service temperature of the corresponding sample. The sample was heated from 30-

1000°C at a heating rate of 20°C/min and its weight loss was recorded and presented in the result section.

2.3.5. Flame retardant testing

Flame retardant testing assesses the propagation of flames under specified fire test conditions. The test conditions are based on the Underwriters Laboratory of United State (UL 94) and are used to serve as a preliminary indication of plastics acceptability for use as part of an appliance concerning its flammability. Based on the material properties to resist fire, the rating system is classified into 2 categories, i.e. Horizontal burn (HB) and Vertical burn (V2, V1, V0). The schematic is shown in Table 3 below. At least 10 specimens with a dimension of 5.0 x 0.5x 0.118 inches are prepared for each test of horizontal and vertical testing.

Table 3

Schematic of UL94 flame retardant test

	Horizontal burn (H-B)	Vertical burn
Specimen setup		
Description	First test (Least flame-retardant UL94 rating)	Second test after the specimen pass the H-B test
Characteristics	Slow burn	Self-extinguishing
Pass requirement	Pass the test if the specimen takes more than 3 min to burn 4 inches.	V-0: self-extinguish within 10 secs after five applications of 5 secs each flame (Best) (No flaming drips are allowed) V-1: self-extinguish within 60 secs (good) (no flaming drips are allowed) V-2: self-extinguish within 60 secs (flaming drips are allowed)

2.3.6. Scratch and wear testing

For the products to serve as countertop or tiles, it is important that the specimen has substantial resistant to scratch and wear. Scratch testing in this study was conducted using

Macro scratch tester as illustrated schematically in Fig. 6A. A stylus with sharp diamond tip was moved over a specimen surface with ascending load from 0 – 100 N with a scratch length of 50 mm. The penetration depth also increased progressively from 0 to 50 mm mark. The penetration depth profile of PGC produced in this study was then compared with commercial natural and engineering stone.

Besides scratch testing, resistant of material to wear is also one of the most important properties. Wear testing evaluates the performance of products over time. The schematic of the wear testing is shown in Fig. 6B using Tribometer. A ruby ball of 5 mm diameter under an applied load of 10 N was used to indent the samples and oscillate from 0 to 50 mm mark for 6000 cycles at 5 cm/s. The depth profile was then measured under profilometer. The intent of wear and scratch testing in this study was to produce data that will reproducibly rank the new materials with the existing products under a specified set of condition.

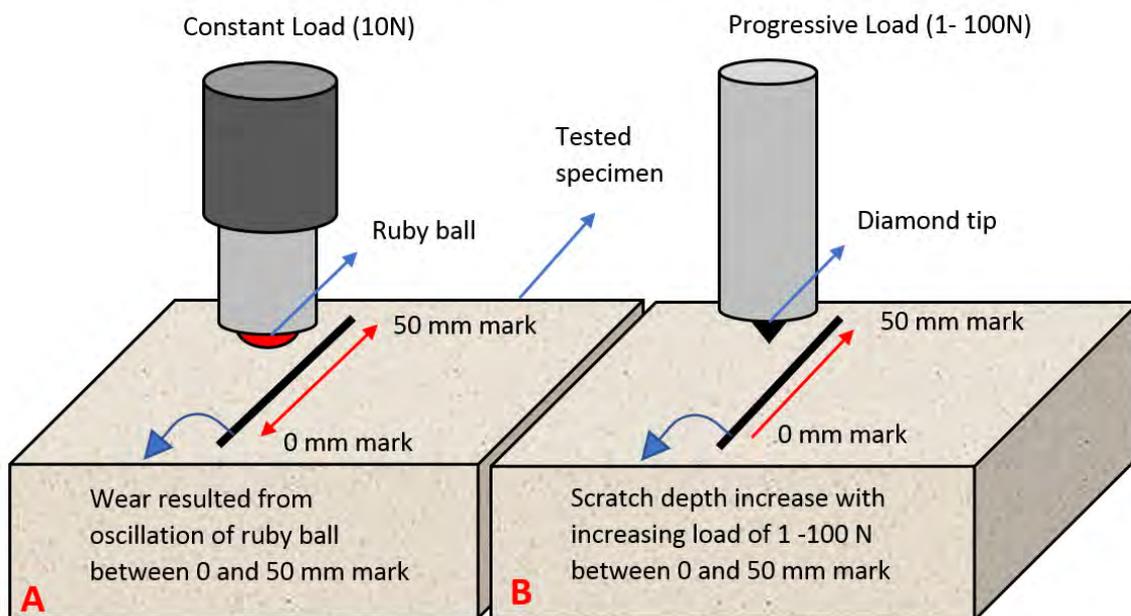


Fig. 6. Schematic of (A) wear resistant test (B) scratch resistant test

3. Results and discussion

The result summarizes the mechanical properties of the produced panels. Effect of the resin-powder ratio, as well as coupling agents on PGCs, were reported in this study. A thorough analysis of the fracture surface of treated and untreated samples was compared by SEM analysis. The goal of this study is to produce recycled panels with comparable mechanical properties when compared alongside with the reference samples.

3.1. Workability and trapped air bubbles

The workability of the pre-cured PGC paste is largely influenced by the viscosity of the resin and glass powder mixture. The goal is to identify an optimal formula for creating a product with desirable mechanical and physical properties without air trapped bubbles. The percentage of resin used was adjusted from 15 to 35%. This range was selected for two main reasons. A mixture of more than 35% resin have lower viscosity and are easily workable but will result in a softer panel. By lowering the resin percentage, the end products are stiffer, imitating a stone-like panel. Secondly, as the percentage resin is the key factor in determining the production costs of the waste glass composite panels, minimising the amount of resin required is essential in delivering a product at a competitive price. It was also found in this study that further decreasing the resin content to less than 15%, however, did not give full coverage for the glass powder.

With 15-35% resin percentage, the mixture was found to have low workability, resulting in a high volume of air-trapped bubbles. To reduce the air bubbles, more precise adjustments of the viscosity and high production pressure were necessary. Viscosity could be altered by adjusting the glass and resin ratio. An acceptable proportion of resin was found to be between 15 and 25% with the optimum ratio of glass/resin for creating a free air-trapped product is 80:20 as shown in Fig. 7. In this viscosity range, the mixture was very stiff but not tacky. It behaved like solid with liquid (wetting) (Fig. 8B) rather than suspension (Fig. 8A). Trapped air could easily be removed by applying high constant pressure and heat. This was because the liquid binder gives sufficient, and not excessive, coverage to the powder; the powder did not float around in the liquid binder. Rather, it acted like wet sand and could be easily compacted using a trowel or pressure. This characteristic allowed the powder particulates to re-arrange, closing the void/ air bubble under high pressure. It also noted that lower viscosity than the range stated would result in a tacky mixture with strong liquid tension (suspension characteristic). As liquid is incompressible, an increase in pressure did not significantly bring the molecules together (Elert, 2017) and air bubbles, therefore, would be difficult to remove, as shown in the mix ratios of 65/35 and 70/30.

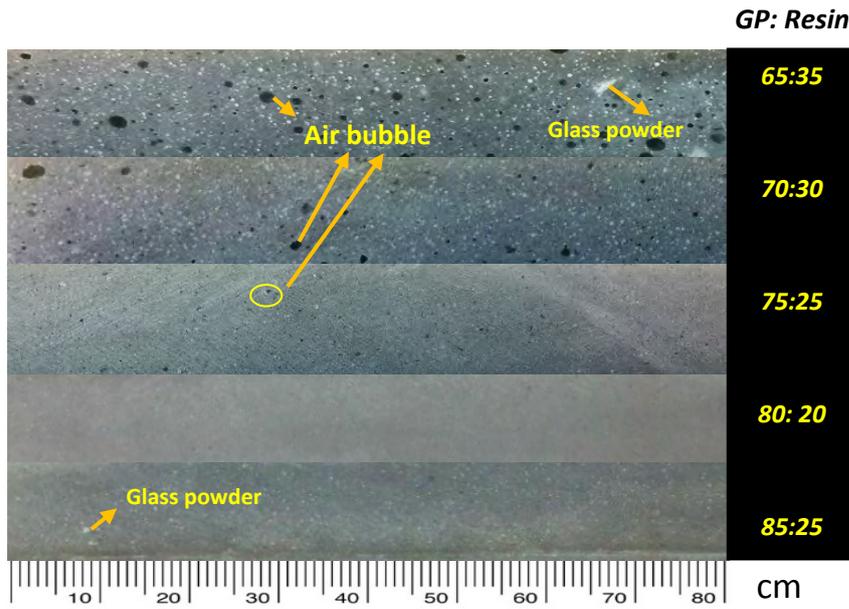


Fig. 7. Cross-section of PGC showing zero air bubble in 75-85% glass powdered concentration

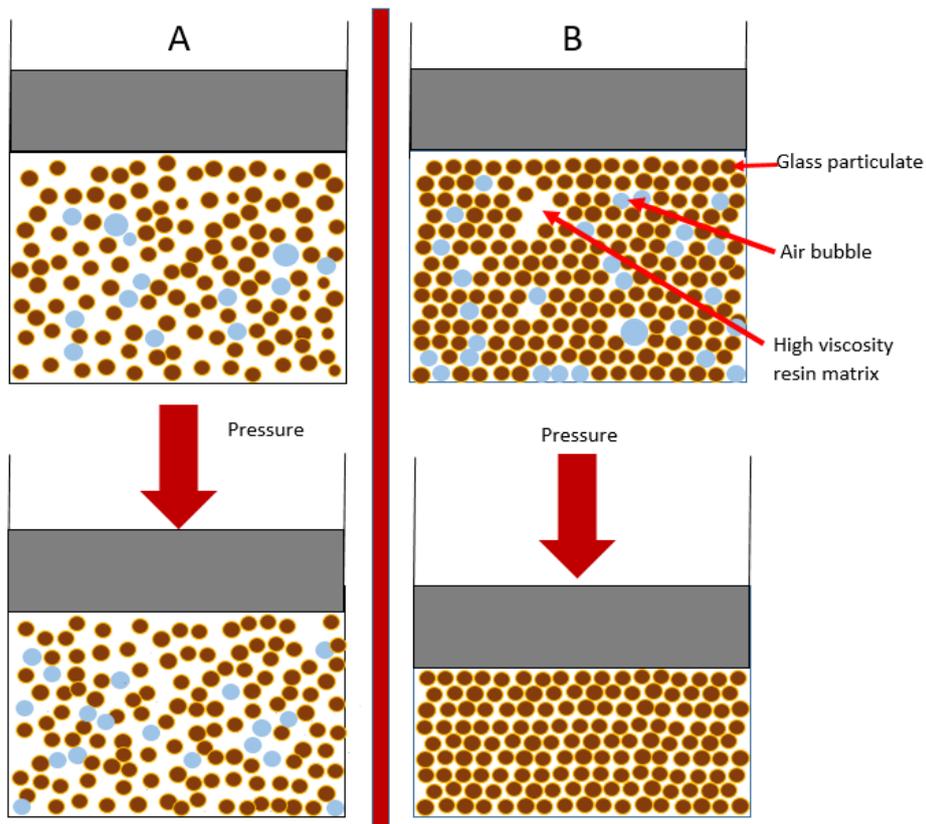


Fig. 8. Schematic of glass powder – resin interaction under compression load at resin percentage (A) smaller than 25% and (B) larger than 25%

3.2. Flexural strength (MOR) and modulus of elasticity (MOE)

Flexural strength, along with density, plays an important role in determining the dimensions of the product produced, especially in table/countertop production in which the beam system is used. Beams span open spaces and are internally self-supporting (National Ready Mixed Concrete Association, 2003). Therefore, higher flexural strength and moderate density are required.

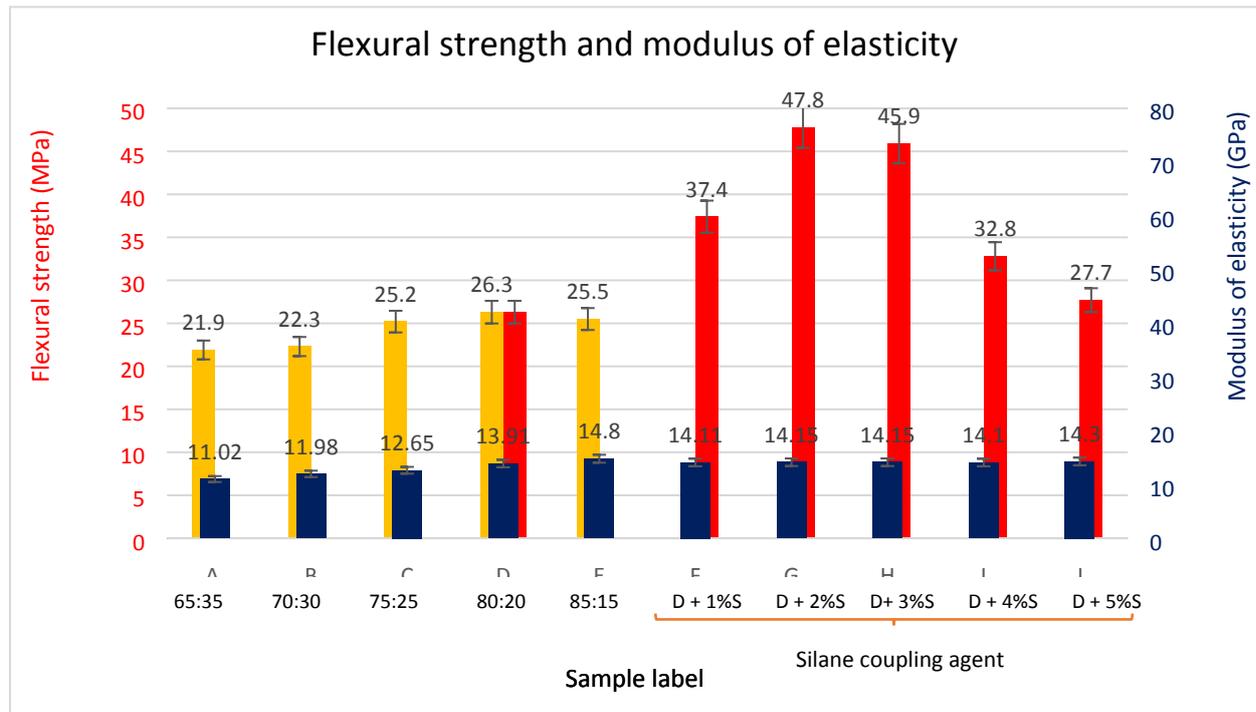


Fig. 9. Flexural strength (MOR) and modulus of elasticity (MOE) of PGC with varying composition and silane coupling agent

3.2.1 Effect of porosity on MOR and MOE

As can be seen from Fig. 9 (A-E), a maximum flexural strength of PGCs without silane coupling agent was 26.3 MPa with glass/resin ratio of 80/20. The improvement might be due to better compaction, smaller porosity and fewer air bubbles in the product compared to other different ratio samples as shown previously in Fig. 7. According to Venkatesh et al. (2016), cracks begin with extreme-sized pores and grow across a specimen, leading, finally, to fracture. The fine pores present in the samples do not seem to affect their ductility and strength significantly. The smaller/negligible porosity of the 80/20 ratio PGC has, therefore, produced a stronger product.

It was also found that there was also a linear correlation between the MOE and glass powder loading. MOE, also known as the flexural modulus is a mechanical property that

measures the composite's stiffness. The higher the value is, the better composite's resistance is to elastic deformation under load or the stiffer the material. Low MOE materials are flexible and tend to deflect considerably under load. To withstand deflection, composites that are placed in a beam system require high MOE. By comparing panel A-E, it was observed that stiffness increase with increasing glass powder content. The increase was mainly due to the addition of high density of glass powder replacing a certain amount of bendable resin binder.

3.2.2 Effect of coupling agent on MOR and MOE

By comparing panel D, F and G, it was apparent that the addition of the coupling agent played a significant role in increasing the flexural strength of the PGCs. Average improvements in flexural strength of more than 40% were observed in these samples, in comparison to control sample (D). The flexural strength increased from 26.3 for panel D to a maximum of 47.8 MPa in panel G, when 2% of silane coupling agent was added. In panel D, interfacial adhesion was weak due to the poor wettability on the surface of glass powder and resin. A weak interfacial region reduced the efficiency of stress transfer along the matrixes and thus resulting in low flexural strength. On the contrary, in panel C, surface modification between these two components was achieved with the addition of silane coupling agent. Wetting of resin on glass powder was more pronounced, resulting in significant improvement in adhesion and compatibility. These increases allowed better stress transfer and thus, significantly improved the bending strength of the PGCs. The improvement was also justified from the SEM analysis which was performed post mechanical testing of the samples.

In [Fig. 10A](#), it was observed that a rough surface with several pores and air gaps occurred at the fracture surface when no coupling agent was added. The rough surface which was due to particle pull-out implied that the bonding between the powder filler and resin was relatively weak (delamination). In contrast, the fracture surfaces of the PGCs samples with the coupling agent ([Fig. 10B](#)) showed massive shear deformation. The strong bonding among all components prevented delamination and encouraged shear yielding before failure. More energy was absorbed by such shear deformation which led to improvements in the bending strength values.

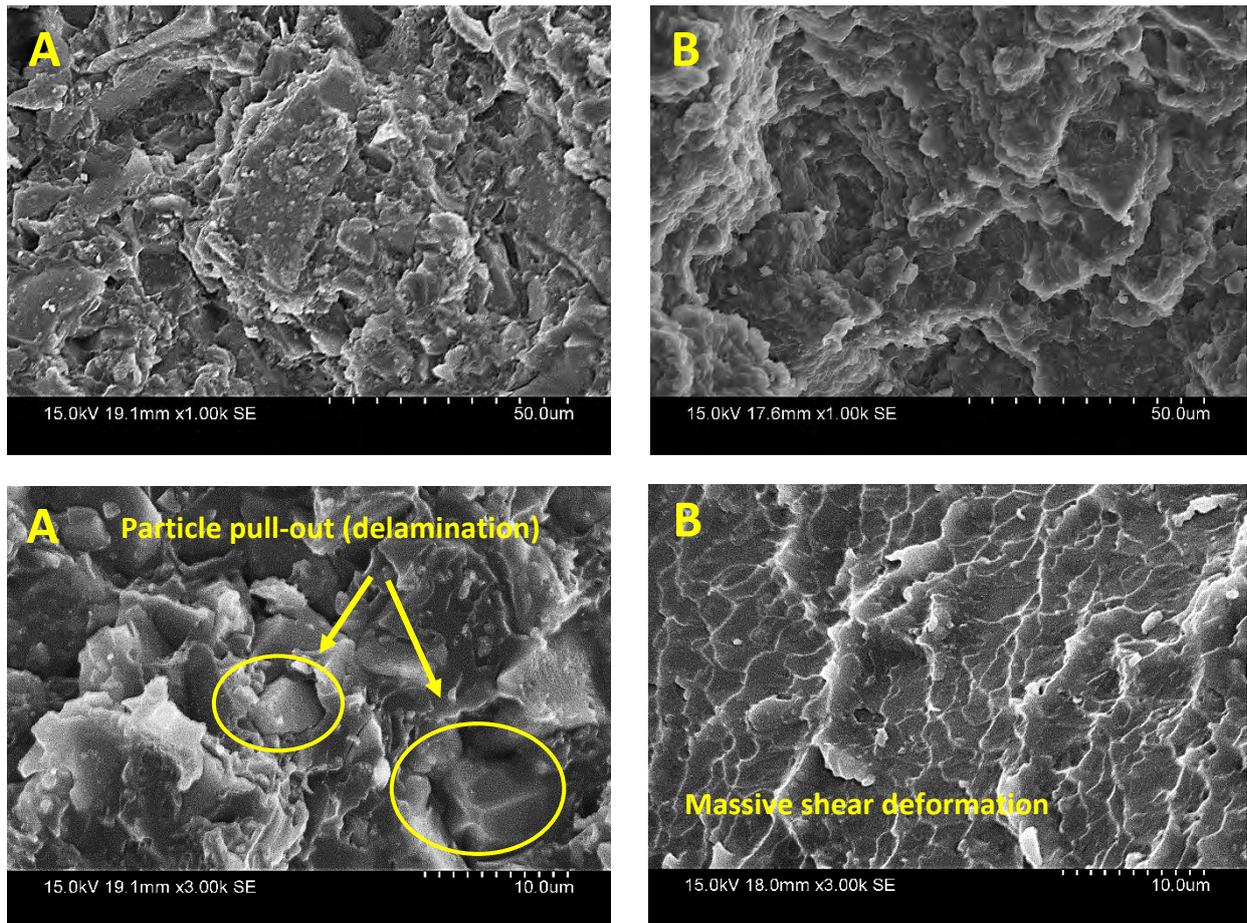


Fig. 10. (A) Delamination of glass bead of PGC without coupling agent (B) Interface modification of glass powder and resin with 2% silane coupling agent

As shown in Fig. 9 (H-J), further additions of coupling agent, however, showed a reverse effect on the MOR. As the volume of coupling agent increased, the surface glass powder was covered by -OH again, leading to poor compatibility and weak interface bonding with the resin (Fig. 3) and, consequently, the mechanical properties of the composite deteriorated.

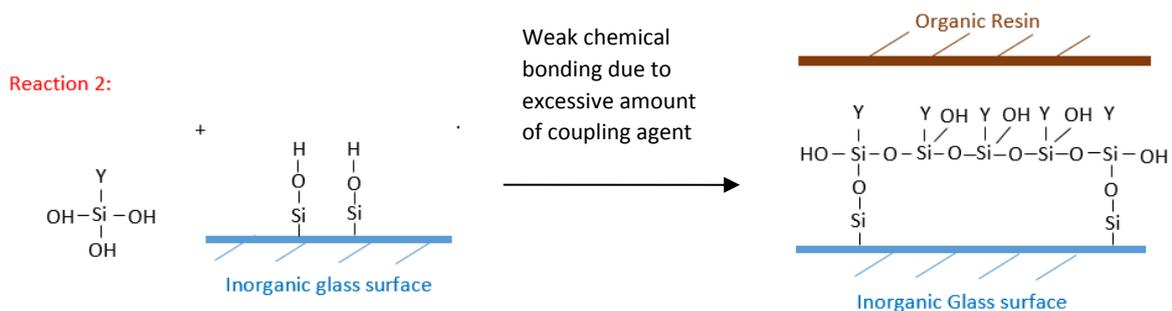


Fig. 11. Weak chemical bonding between glass powder and resin due to excessive amounts of coupling agent

3.3. Compressive strength

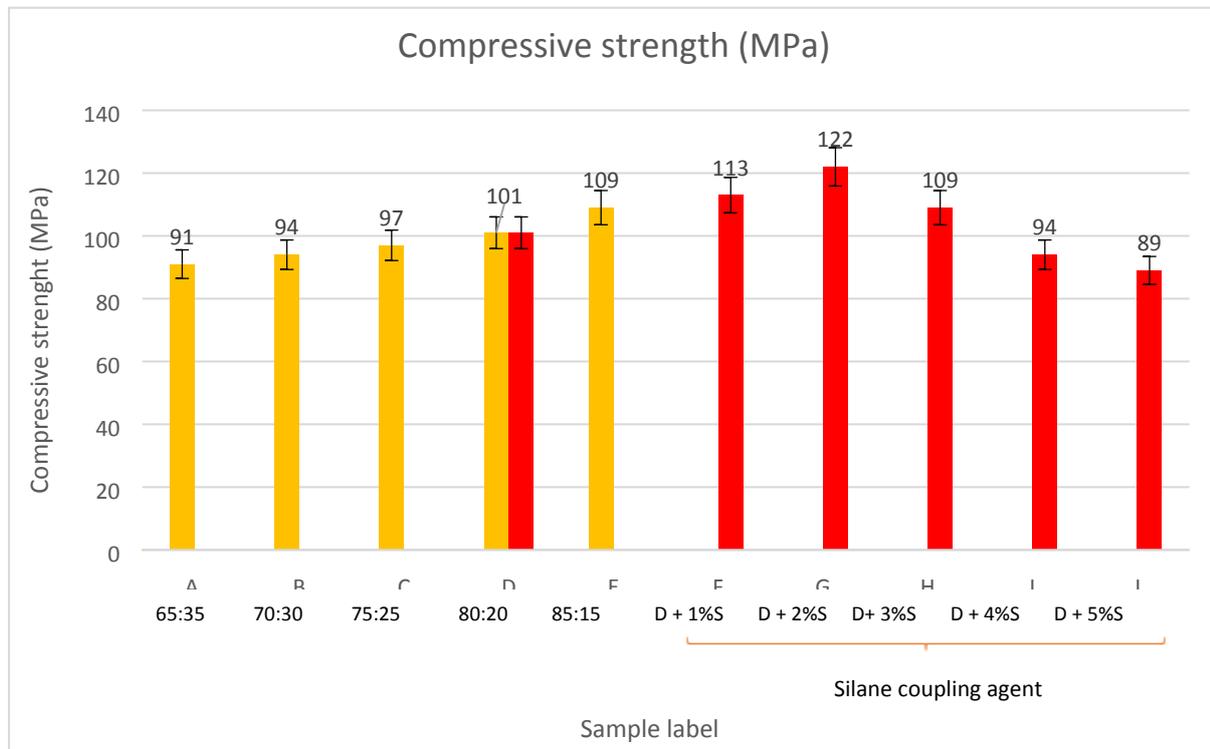


Fig. 12. Compressive strength of PGC with varying compositions and with/without silane coupling agent

Fig. 12 (A-E) shows the compressive strength of PGCs increased from 91 to 109 MPa with increasing glass content from 65 to 85 percent weight. In all tested samples, the higher the glass content was, the more difficult it was for a crack to propagate, resulting in higher compressive strength. The improvement might also be due to better compaction, smaller porosity and fewer air bubbles in the product.

By comparing panels F – J with D, it could be seen that the addition of coupling agent resulted in improvements in an average compressive strength of panel D (80:20, without coupling agent) from 101 to a maximum value of 122 MPa in panel G when 2% of silane coupling agent was added. The increase was due to the established bonding capacity between the resin and glass. However, further additions of silane coupling agent had a reverse effect on compressive strength, as a result of the phenomena explained earlier in case of flexural strength.

3.4. Comparison with the standard

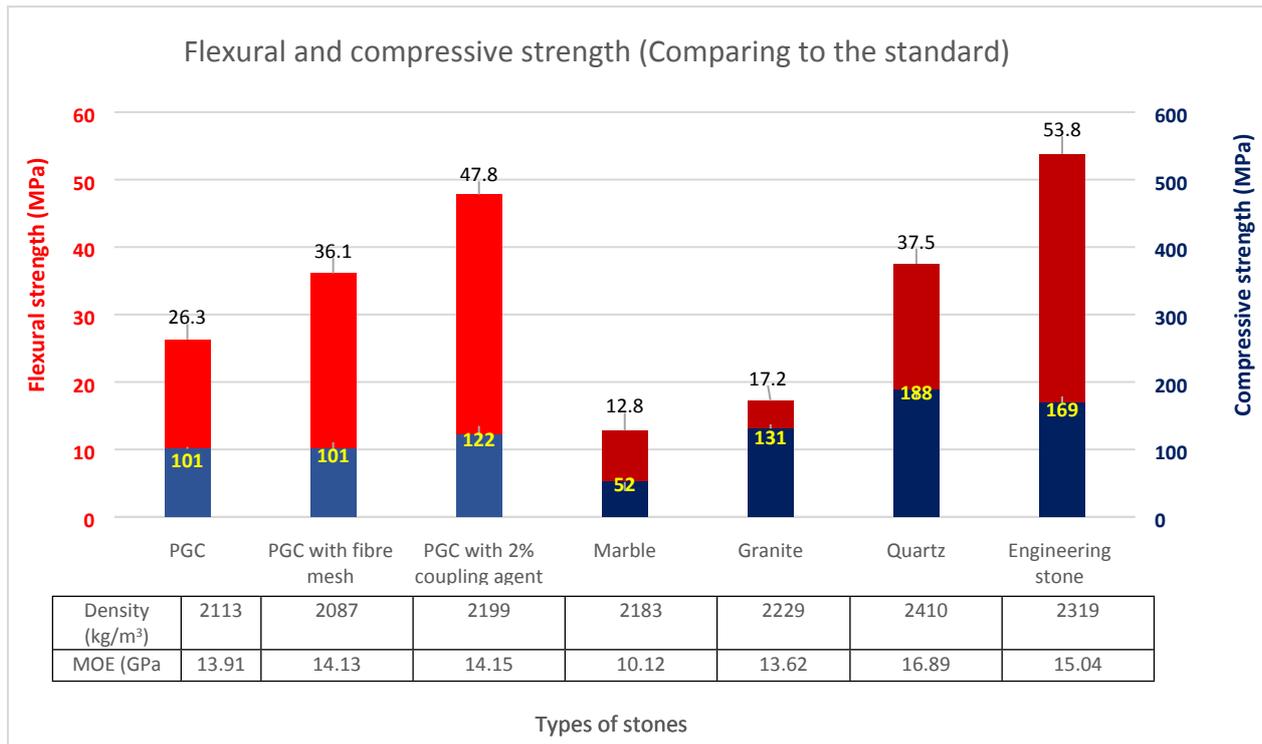


Fig. 13. Comparison of mechanical properties of PGCs with the natural and engineering stone

By comparing the PGCs samples produced with natural stone (Fig. 13), it was found that PGCs offered superior performance in both flexural strength and density. In terms of flexural strength, PGCs with a silane coupling agent were three times more resistant to bending than natural stone (marble and granite) and exhibited comparable properties to Quartz and engineered stone tested. It is important to note that although both natural granite and quartz was composed from the same crystalline SiO₂, the natural granite collected in this study had larger particle size (2-4 mm) compared to Quartz which might degrade its bending strength significantly. Even without the addition of a coupling agent, the flexural strength of the PGC samples was adequate for countertop or tabletops applications. If thinner sections of the PGCs are required, a sheet of fibreglass can be added as an alternative to the coupling agent. The addition of fibre glass mesh and silane coupling agent to the PGC improved flexural strength by up to 37% and 80%, respectively. Besides flexural strength, the densities of various PGCs were also slightly lower, compared to natural or engineered stone. This was due to the use of 20% resin which has a density of 1.83 g/cm³.

The stiffness of PGC and engineering stone was also found to be higher compared to marble and granite stone. The stiffness of natural stone is affected by many factors, i.e. the chemical bond that made the stone, impurities, defect as well as the structure (Rong et al. 2013).

Quartz, granite, glass and engineering stone which are composed of SiO_2 have stronger bonding compared to CaCO_3 in marble stone, which affects its stiffness. In granite stone, impurities such as feldspar, mica, amphiboles and other minerals might weaken the strength as well as the stiffness.

It can also be seen that the stiffness of engineering stone and Quartz are 16.89 and 15.04 GPa respectively. Although Engineering stone was made from the same materials as natural Quartz, the ductile properties of resin addition in engineering stone might be the result of the decrease in stiffness. The decrease was, however, not very significant due to a small amount of resin added and higher amount of powder filler content.

Further observation of Quartz and glass were also investigated in this study. Unlike Quartz which has strong covalent bonds that hold the silicon and oxygen in arranged covalent structure, the addition of Na_2O structure in glass disrupts the structure of quartz by adding oxygen atoms more than those required for an interwoven tetrahedral structure (Jiang, 2014). The bonding in a glass is slightly inferior to Quartz based stone and thus affecting the stiffness. The stiffness of glass, however, was still relatively high compared to marble and granite, with a small decrease of MOE due to resin addition in PGCs. Regardless of the variation in the MOE value, all the samples tested were very stiff and underwent brittle failure with minimum deflection during testing.

The combination of low density, high stiffness and flexural strength in both PGCs and engineered stone, when comparing to natural stone products, can be expected to facilitate the production of thinner PGC countertop slabs with longer spans. This creates a new sustainable solution in providing path-breaking building product which will lead dematerialisation.

In terms of compressive strength, Fig. 13 shows the PGCs are inferior to Granite, Quartz and engineered stone. The addition of fibreglass sheeting increased the flexural strength but showed negligible improvement in the compressive strength. Likewise, the addition of a coupling agent increased in compressive strength of only about 20%. However, although the compressive strength values were not as high as granite and engineered stone, they were above the compressive strengths required, and adequate for the intended applications. By comparison, high-performance concrete has compressive stress in the range of 70- 80 MPa (National Ready Mixed Concrete Association, 2003).

3.5. Scratch resistant test

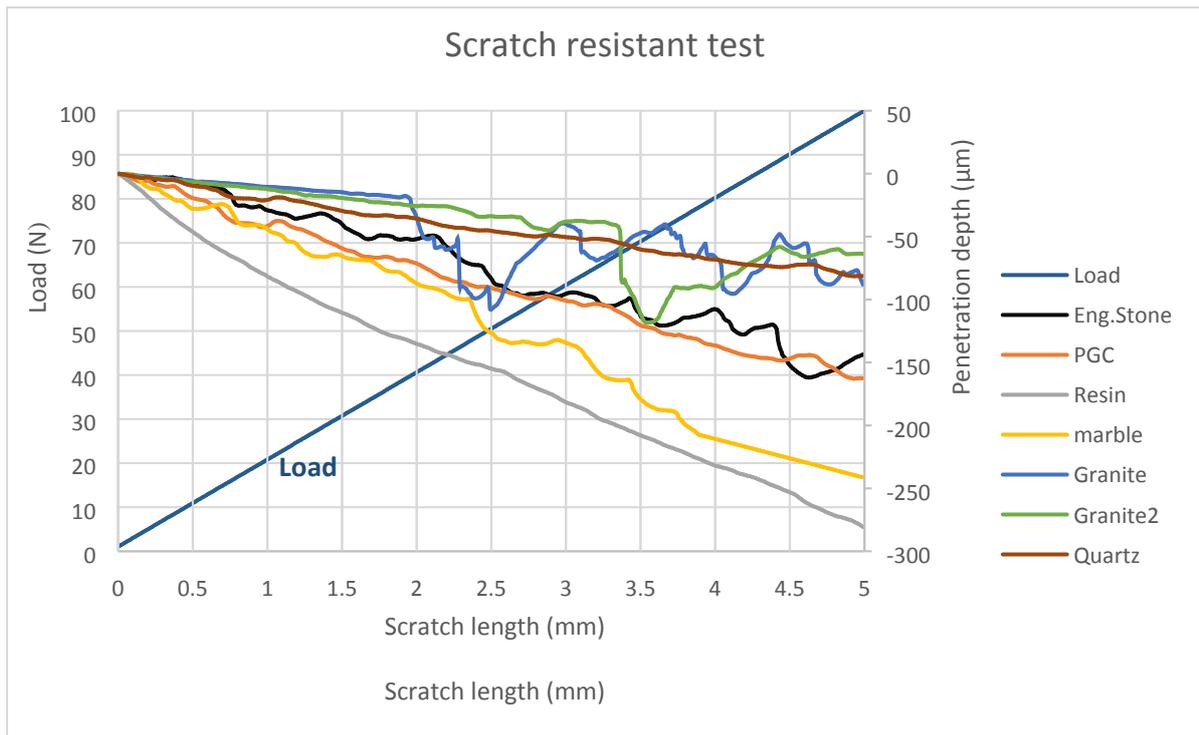


Fig. 14. Penetration depth of tested samples

Fig. 14 illustrates the penetration depth of the tested samples at increasing load of 1- 100 N within 5 mm scratch length. It was observed that the penetration depth in PGC increased linearly with a load from 0- 160 μm . The value was quite comparable with engineering stone with depth of 0 – 150 μm . The slightly less scratch resistant values in PGC was due to the nature of glass which has a lower hardness (Mohs hardness: 5.5) compared to engineering stone which constitutes mainly of Quartz powder (Mohs hardness: 7) (Mineralogical Society of America. 1997). Furthermore, by comparing resin alone with PGC, it was also observed that the scratch-resistant increase nearly two-fold with the addition of glass powder filler. All the synthetic stones produced, however, showed inferior performances in comparison to natural granite and Quartz but demonstrated higher scratch resistant value than marble. Quartz and granite had penetration trend line of - 15 $\mu\text{m}/\text{cm}$ and - 12 $\mu\text{m}/\text{cm}$ respectively. This was due to the harder crystalline SiO_2 fillers that made the materials. Regardless of the loading rate, some impurities in granite, however, resulted in deeper scratch depth.

3.6. Wear testing

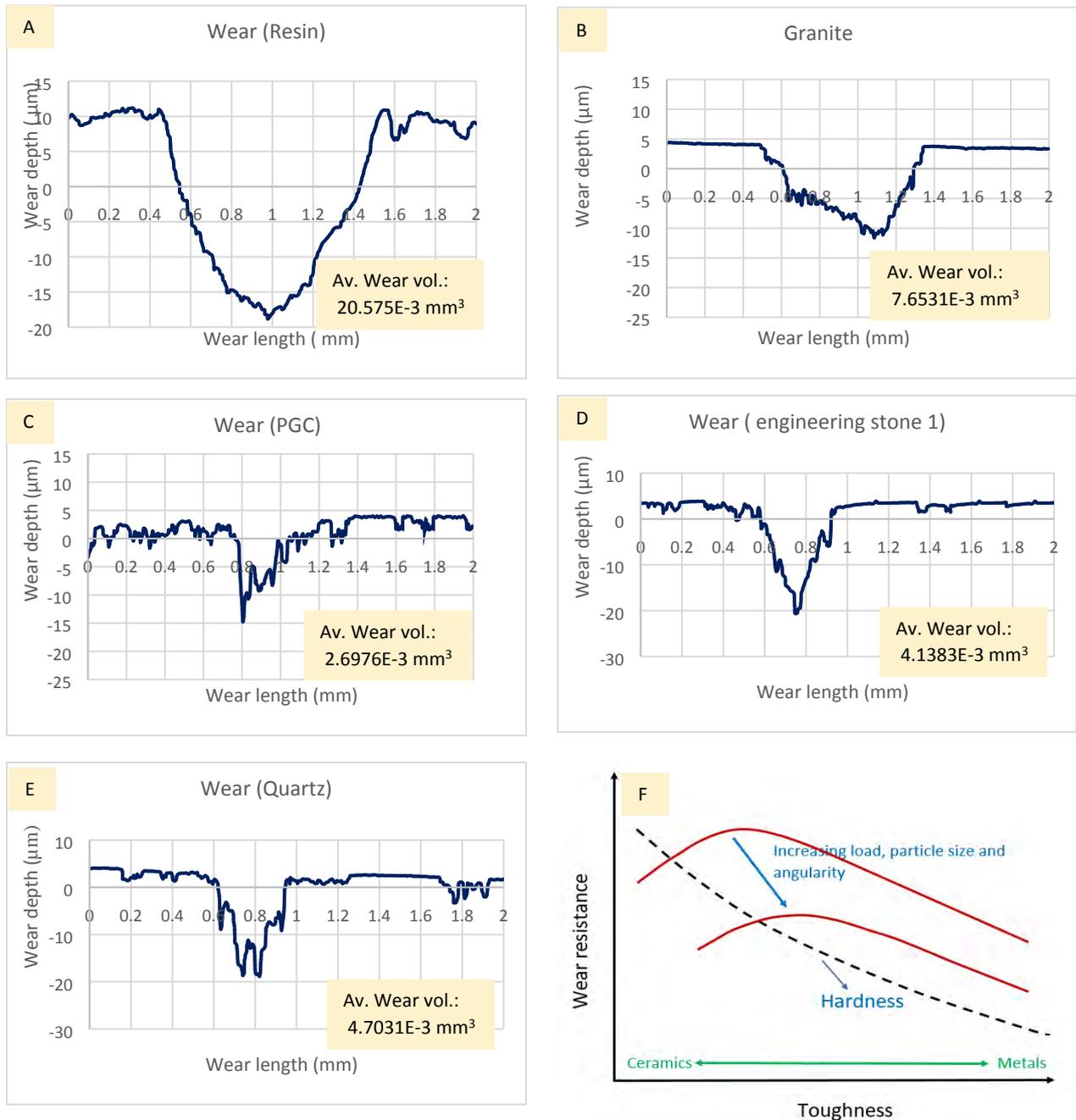


Fig. 15. Wear profile of tested samples (A-E); (F) Correlation of wear resistant with hardness

Fig. 14 illustrates the penetration depth profile of the tested samples under wear testing for 6000 cycles at 10 N load. The graph of the wear was drawn by using profilometer. It was then followed by plotting the data in Excel and transfer to AutoCad to get an accurate measurement of wear depth area. It could be observed in the graph that PGC had the least wear with wear volume of $2.6976 \text{ E-}3 \text{ mm}^3$. The better performance of PGC in comparison

to engineering stone (wear volume 4.1383) was due to the use of finer powder filler ($< 108 \mu\text{m}$) in PGC production. In contrast, the particles size of engineering stone was shown under an optical microscope in Fig. 16 to be about 0.05 mm in diameter. Larger particles cause more extensive wear as they carry more kinetic energy (University of Cambridge, 2016). Similarly, a natural Quartz and granite which consist of larger angular aggregates showed inferior performance with wear area of $4.7031\text{E-}3$ and $7.6531\text{E-}3 \text{ mm}^3$ respectively compared to both the artificial stones. The size and shape of natural SiO_2 stone affect the rate of wearing significantly with angular particles cause greater wear than round particles (University of Cambridge, 2016). The natural quartz was made from finer particles (0.1-0.5 mm size) compared to granite with particle size ranging from 2 to 4 mm, which result in much better wear performance in Quartz. Higher impurities in granite compared to Quartz stone might also be the reason of the inferior performance of granite. Besides size, shape and impurities, hardness also plays an important role in wear. Brittle material like ceramics and natural stone usually suffers wear by brittle fracture with ductile materials like metal, plastic and resin suffer substantial wear by plastic deformation (University of Cambridge, 2016). The resin used in this study was ductile and produced wear volume of $20 \text{ E-}3 \text{ mm}^3$ under the same experimental condition, nearly three-fold compared to all the tested samples. According to research conducted in University of Cambridge (2016), a maximum wear resistance arises through a combination of intermediate values of hardness and toughness as shown in Fig. 15 F. PGC and engineering stone which consisted of a combination of ductile resin and brittle powder had therefore performed better in wear. Wear-resistant of marble stone was not reported due to excessive wear at only 1000 cycles and was considered fail under the testing condition.

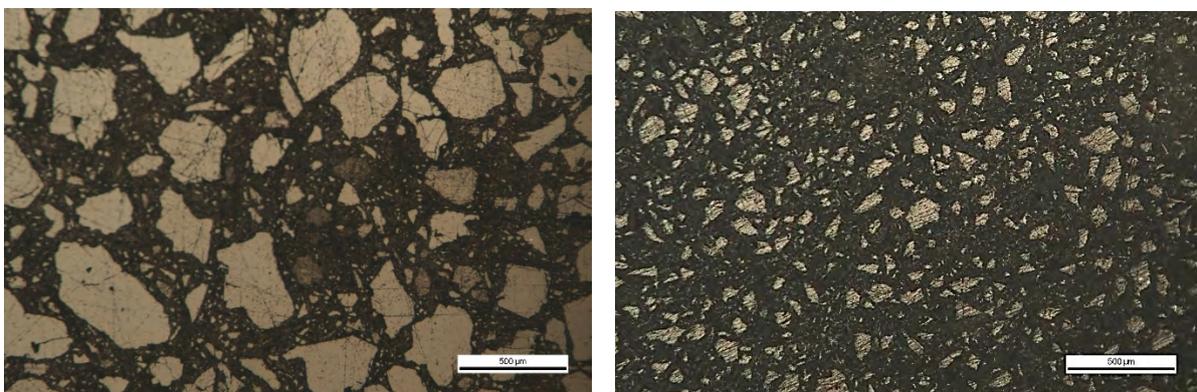


Fig. 16. Particle size distribution in (A) engineering stone (B) PGC

3.7. Water absorption

Fig. 17 summarises the water absorption of the tested samples. It was observed that the PGC samples without coating show average water absorption of around 0.003%. An improvement to 0.00112% was observed with the addition of stone sealer. The stone sealer used in this study was granite gold sealer which is non-toxic and safe for food preparation surface. After the addition, the value is comparable to that of coated natural stone and engineering stone existing in the market. Without the coating, marble and granite are porous and were reported to absorb nearly 0.06 and 0.04% of water respectively (Kessler, 1919). The uncoated values of PGCs were found to be much lower compared to the natural stone. No significant improvement in water resistance was observed with the addition of coupling agent and fibre glass mesh. In this study, immersed specimens had also been tested under flexural and compression test. However, no significant differences were observed due to a negligible amount of water absorbed by the specimens.

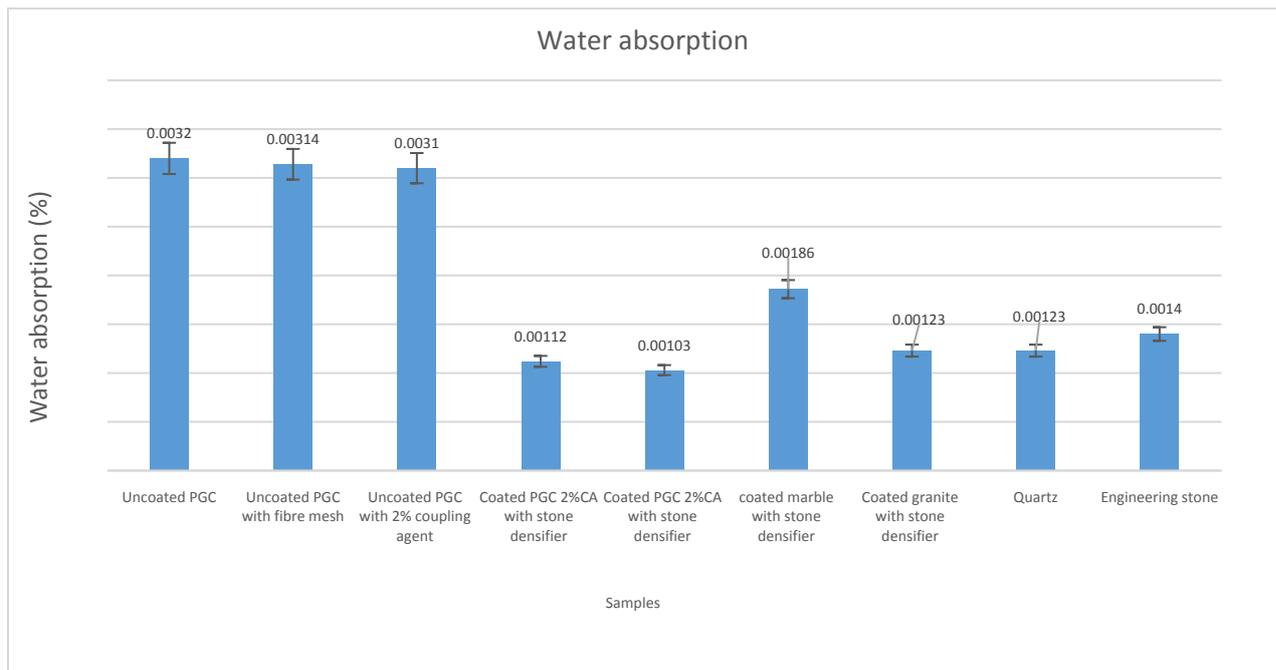


Fig. 17. Comparison of water absorption of uncoated PGCs with the natural and engineering stone

Dimension stone countertop manufacturers usually offer additional coatings; such coatings can similarly be applied to give extra protection to the PGCs. Polyurethane (PU) or polyasparthic coating about 0.1 mm thick provided extra resistance to water, stains and Ultraviolet (UV) in the final coated PGC product. However, a light sanding of the uncoated

PGC surface is necessary before applying the polyurethane coating to prevent delamination, as shown in Fig. 18 below.

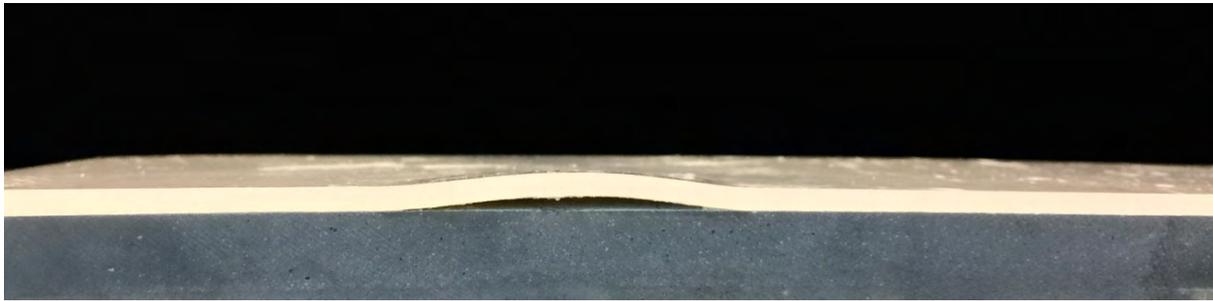


Fig. 18. Delamination of polyurethane coat in PGCS

3.8. Thermal degradation and scorch resistant testing

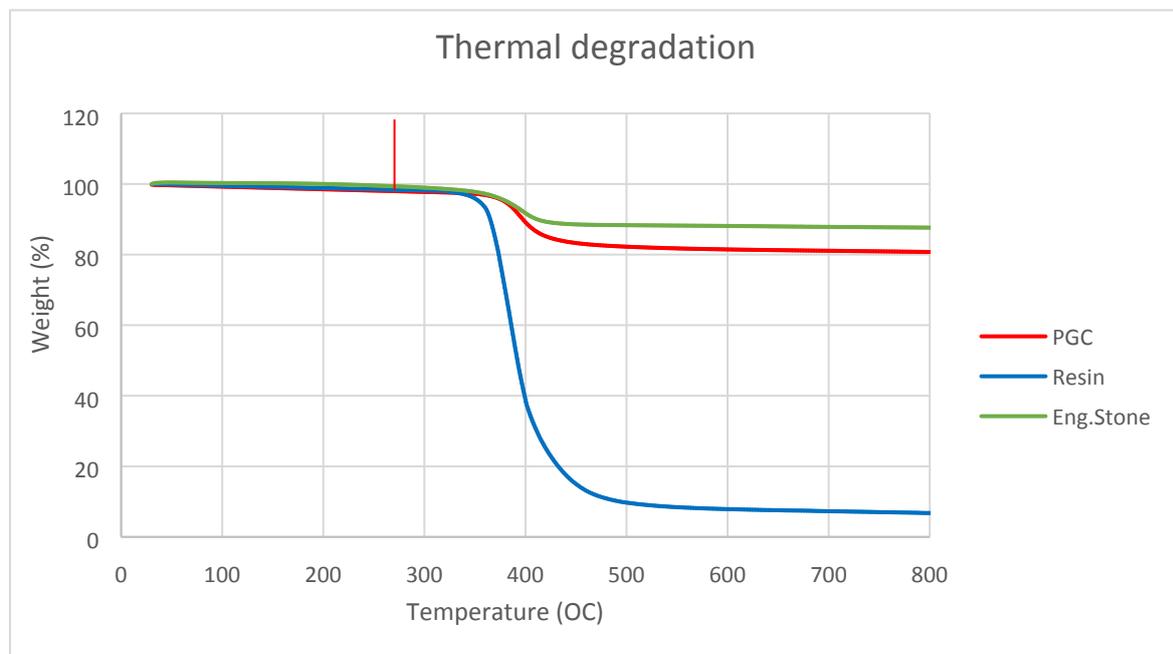


Fig. 19. Thermal degradation of artificial stone and resin

Thermal degradation analysis is an important assessment to estimate the maximum service temperature of materials, especially polymer which tend to lose its mechanical strength at relatively low temperature. The degradation was measured by using Thermogravimetric analysis (TGA). PGC and engineering stone consist of a polymer binder. At elevated temperatures, the components of the long chain backbone begin to break apart. It can be seen from the Fig. 19 that PGC and Engineering stone began to degrade at around

270°C with maximum degradation occurring after 350°C which fell at the same degradation temperature as the resin binder. PGC was observed to have more weight loss compared to engineering stone with loss of 18% and 12% respectively. This might be due to the use of a smaller amount of resin in engineering stone (7%) compared to PGC (20%). Regardless, the service temperature of these two materials fell on the same category.

Besides TGA, scorch testing was also conducted in this study as shown in Fig. 20. A hot steel with temperature ranging from 200 to 1000°C was placed on top of PGC sample for 30 minutes. No apparent defect was observed in PGC at a temperature below 400°C. However, similar to engineering stone which was made from resin binder, it was recommended to always put a trivet or barrier between a hot material and PGC surface. As shown from TGA analysis, strength might be compensated at a temperature above the degradation temperature. Moreover, the different thermal expansion in glass powder and resin, although small, along with contraction and expansion of heated and unheated area might result in crack of the countertop in the long run. Furthermore, it could also be observed that white spot start to occur at a temperature as low as 450°C, and turn into burn mark at a temperature of 700°C.

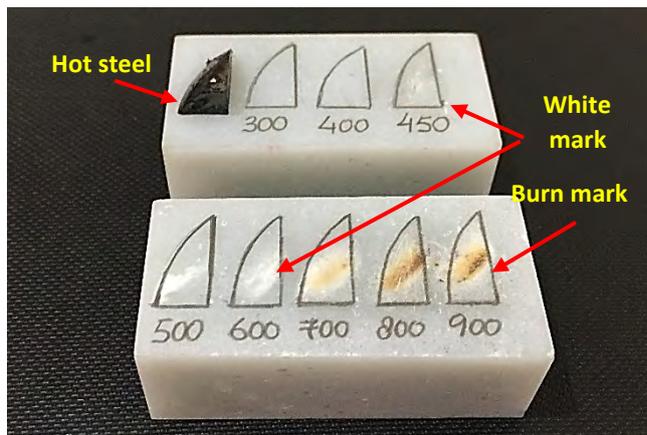


Fig. 20. Scorch test of PGC at 8 different temperature (Unit: Celsius)

3.9. Flame retardant testing

Table 4.

Flame retardant testing of different stone composite.

Samples	Horizontal burn	Vertical burn (Average total flaming combustion)
Resin	Pass (12.7 mm/min)	Fail

PGC	Pass (self-extinguish)	V1 (< 225 secs)
PGC with 2% CA	Pass (self-extinguish)	V1 (< 216 secs)
Commercial samples		
Marble	Natural stones do not contain polymer binder, therefore they passed all the required flame retardant test.	
Granite		
Quartz		
Engineering stone	Pass (self-extinguish)	V1 (< 210 secs)

PGCs consist of resin binder that is categorised as a plastic material. It is imperative that the composite produced have reasonable resistant to fire, especially when it is used for a kitchen application. The flame-retardant testing is based on Underwriters Laboratories of the United States (UL 94) and is used to serve as a preliminary indication of plastics acceptability for use as part of a device or appliance with respect to its flammability. The rating system is categorised into 6 types, i.e. HB (least flame retardant), V2, V1, V0, 5VB to 5VA (most fire retardant). Most of the tested samples passed the horizontal burn test with PGC and commercial engineering stone showed self-extinguish properties when laid flat. This test was particularly important considering the slab produced would serve horizontally as countertop, tiles and table. The cured resin itself also had considerable resistant to flame spreading of 12.7 mm/min. Unlike thermoplastic which tends to soften and flow at high temperatures, thermosetting resin does not soften but undergo localised surface charring which impedes the spread of flame. Furthermore, it was observed from the table that fire-resistant property increased with the addition of glass powder. The improved fire resistance observed was largely due to the non-flammable and non-combustible nature of glass powder, which provided temporary barriers to the flame as it spread along the WPCs. Furthermore, the minor amount of sodium silicate in the glass powder might also play a considerable role in these improvements. Sodium silicate has been widely used as passive fire protection. It has a synergistic effect on the intumescent flame retardant (IFR) when exposed to an open flame. It increases in volume and decreases in density, forming char at higher temperatures. The char is a poor heat conductor, preventing the fire from spreading further (Slimak and SLimak, 2011). From the graph, it could also be observed that the PGC produced passes the vertical burn test (V1) with total combustion time for 5 times not exceeding 250 seconds and no flaming drips were observed. The result was comparable to the commercial engineering stone in the market. Natural stone like marble,

granite and Quartz do not contain polymer binder, therefore has excellent flame-retardant properties.

3.10. Improving the aesthetic look of PGC

A range of colours, effects and ‘looks’ for the PGCs was developed in the lab using waste materials, coloured stone powder and synthetic liquid pigments, as shown in Fig. 21 below. Sample A was made by using 0.2% carbon powder and 1% of white liquid pigment. The swirling effect was the result of the partial mixing of the coloured materials with the pre-mixed glass powder-resin mixture. Similarly, the blue product (Fig. 21 B) was made using the same process, but with 1.2% blue and white liquid pigment. Other samples, such as Fig. 21 C, had been made with the addition of glass aggregates. Additives such as copper powder from e-waste, Quartz stone fragments, sea shell from food waste can be embedded in the mixture before casting. These samples proved that other waste materials could also be absorbed into the PGCs to improve their aesthetic look, providing a cost-effective ‘waste-derived’ product that is comparable to natural dimension stone.

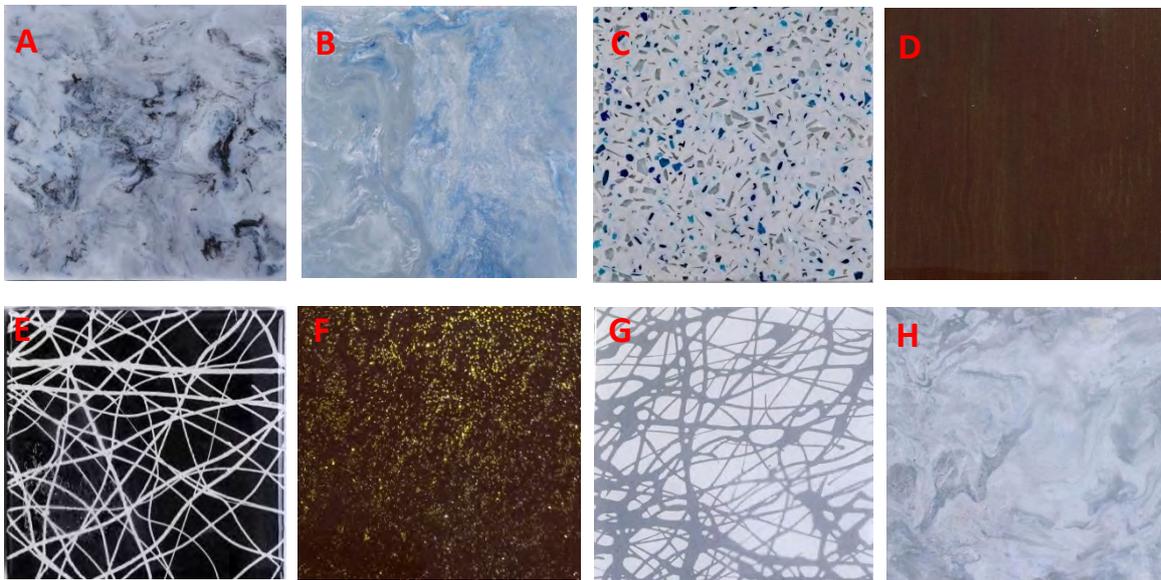


Fig. 21. (A-C) PGC with colour pigment added

4. Conclusion

This study demonstrates the successful manufacture of high-performance artificial construction slabs from waste glass powder filler. With high compression pressure, heat and an optimal mixing ratio, a densified polymeric glass composite (PGC) panel with a flexural strength of 26 MPa, a compressive strength of 101 MPa and water absorption below 0.0032% was produced. The flexural and compressive strength of the PGCs was found to improve significantly to 47.8 and 122 MPa, respectively, with the addition of a 2% coupling agent. The increases were due to the improved adhesion between the resin and glass powder. However, further additions, from 3-5%, showed the reverse effect. As more coupling agent was used, the surface glass powder became enwrapped by -OH again, leading to poor compatibility and weak interface bonding with the resin, leading to a deterioration in the mechanical properties of the composite. Further investigation also showed the addition of a coupling agent from 0-5% showed negligible improvement in both water absorption and hardness. As an alternative to a coupling agent, a sheet of fibreglass was successfully tested as a cheaper means of improving the flexural strength of the PGCs, particularly for thin, narrow slabs. Wear, scratch, thermal decomposition and fire retardancy of PGC were also investigated and are found to be comparable with engineering stone. To improve the aesthetic appeal, other waste such as coloured stone, copper powder from e-waste and tinted broken glass can also be added. These results demonstrated that a high quality and versatile product, comparable to engineered stone, could be produced primarily from waste glass powder. Given the emerging global challenges posed by waste and the need for low-cost, locally available inputs for manufacturing, this process offers an important alternative for the waste glass that cannot be remelted in conventional recycling processes. As such, it has the potential to deliver significant economic and environmental benefits to almost any community, given the relatively simple manufacturing process and the wide availability of waste glass.

Limitation and future studies

PGCs were considered promising and potentially sustainable materials. One complication is that the waste streams of PGCs might be more complex than the virgin materials in that they often contain more than few components. Internal recycling from off-cut panels has been done in the laboratory. The PGCs were crushed back into 1-2 cm aggregates and mixed with resin and coupling agent to form granite-like slab with superb mechanical

properties. For the end-of-life composite panels, however, focused researches are required. Factors like degradation of PGCs due to repeated processing cycles and environmental exposure might complicate the recycling process. These are not insurmountable issues but will require a thorough and coordinate research approach. Future studies might also include an investigation on the effect of different waste filler on the production of resin-powder composite and in-cooperation of glass aggregate in PGCs.

Acknowledgements

The research was financially supported by CRC low carbon living (RP10022). Special thanks to Dr. Irshad Mansuri for providing technical assistance and Mr. Bill Joe who plays a vital role in assisting with all the mechanical testing. We would also gratefully acknowledge the support provided by Sigrid Fraser from Mark Wainwright analytical centre (MWAC), Sarah Kelloway from XRF and George Yang for sharing their knowledge and Ms. Louise Williams for editing the manuscript.

List of abbreviations

ASR	Alkali Silica Reactivity
ASTM	American Society for Testing and Materials
CSH	Calcium Silicate Hydroxide
GP	Glass Powder
PGC	Polymeric glass composite
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MWAC	Mark Wainwright Analytical Centre
PU	Polyurethane
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric analysis
UL	Underwriters Laboratories
UV	Ultraviolet
XRD	X-ray diffraction
XRF	X-Ray Fluorescence

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Highlights

- A novel glass based composite from local waste materials is proposed.
- Glass powder filler is examined and combined with proprietary resin to produce polymeric marble – like stone composite.
- The prototypes demonstrated superior mechanical performance and minimal water absorption.
- The aesthetic look and mechanical properties of the composite are comparable to granite, marble, Quartz and engineering stone existing in the market.