Adhesion test for epoxy reinforcing using waste materials applied on concrete surfaces

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Abstract. There are several methods currently used to protect concrete; among the most prominent of these is surface coating. Different types of materials, as well as various coating methods, are used for the purposes of protection and to strengthen the concrete surface; however, universally agreed criteria for the selection of these materials has not yet been established. This work thus aims to investigate the effects of reinforcing epoxy by using waste material (glass or porcelain) to prepare particulate composites as a form of coating material. Pull-off adhesion tests using controlled concrete specimens are one of the most common methods used to reveal the durability of surface coating systems. This method was thus also adopted for the current work. Samples with and without filler were exposed to hardness and pull off tests to determine the effect of adding the filler content to the epoxy in terms of variation to its properties. Epoxy was reinforced with filler with a 90-micron particle size. Composites of epoxy with varying percentages (0 to 40 wt%) of filler were prepared using a hand lay-up method. The experimental results showed that the addition of waste filler promotes adhesion properties. The 40% percentage of porcelain in the epoxy the largest effect on pull-off strength and hardness tests, reaching 16.12 MPa and 98.62, respectively. For glass composites, the highest hardness result was 97.92 for the 40% percentage. The final results showed a general improvement in the adhesion properties of composites when glass and porcelains were added, with the porcelain results being much better than those of glass. These results achieved the objective of the current work in terms of developing a better coating material for concrete substrates used for construction applications with reduced costs.

Keyword: WG (waste glass), porcelain, epoxy resin, pull-off, adhesion

1. Introduction
Surface concrete protection has undergone several developmental phases in the search for improved new coating processes using different materials, with many application methods trialled with a view to strengthening. The procedures for selecting the materials for concrete coating must also take into account the mechanisms of deterioration that can be carefully diagnosed from the structural conditions [1]. For example, in cases of salt damage, any restoration strategy must take into consideration the corrosion circumstances and the conditions of deterioration, in order to specify the indications, such as (1) removal of permeated chloride ions, (2) a penetration block of chloride ions, moisture, and oxygen, and (3) a corrosion-control method (coating or potential control) [1]. The process of determining the best system and coating materials can thus be obvious, when such choices result from the limited durability data available. Recently, many new industrial applications have emerged, such as construction use of polymer composites instead of metals; this has resulted from the fact that the former have several advantages over the latter in terms of low cost and density, high strength-to-weight ratios, and chemical resistance, as well as less risk of damage to equipment during processing. These are in addition to the good mechanical criteria of these polymer composites [2].
Mechanically, the reinforcement and enhancement of mechanical polymer properties are efficiently carried out by using fillers. In terms of impact, adhesion, and tensile properties, so-called inert fillers may still have an influence on characteristics of the compound, in addition to generally reducing the cost. Specifically, they can increase the compound density, reduce shrinkage, and increase hardness [3]. Commonly, protective coating materials such as epoxy come with a high cost to adhesion ratio. Hence, in order to reduce costs and improve adhesion, epoxy is enhanced with waste materials (waste glass and porcelain) in this work. The resulting composites, with the addition of porcelain first and glass second, were submitted to hardness tests, revealing better readings for hardness that unmodified epoxy. Thus, it is suggested that composites of these particulates are used as coating material. To prove the validity of such material as a coating, a pull-off test was also carried out. Coatings with high values of modulus, small pre-existing cracks, and other mechanism of dissipating energy are likely to increase the pull-off values in materials. This increase in the mechanical modulus and the measured pull-off can thus be achieved by adding filler to the polymer, due to the increase in the density of particulate composites [4].

2. Experimental Procedure

2.1. Materials System
2.1.1. Epoxy
The matrix material used in the present study was epoxy type EUXIT 50\(^1\) non-pigmented, which consists of two components, liquid epoxy resin and formulated amine hardeners. It was supplied by SWISSCHEM Company, Switzerland, and the required ratio is 3 resin: 1 hardener.

2.1.2. Waste Glass (WG)
The source of waste glass used in this work was window waste featuring Turkish-made glass collected from local window glass vendors. The thickness of the glass was 4 mm. After collection, the WG was cleaned, crushed, and sieved in order to obtain a particle size of 90 microns. Many particle sizes can be used to reinforce epoxy; however, the chosen particle size in this work was 90 microns for both glass and porcelain, which were sieved to obtain this particle size. This particular size was selected to ensure good distribution in the epoxy without precipitation, as coarser particles (larger than 90 microns) may settle in the epoxy due to gravity. The physical properties of crushed WG are shown in Table 1.

2.1.3. Porcelain powder
The porcelain powder used in this research was collected from construction waste material remaining on building sites; after they were brought to the lab, samples were crushed using a Jaw crusher then powdered using a HERZOG pulverizing mill HSM, before being sieved using an auto sieve shaker to obtain particles of 90 microns in size. The physical properties of the porcelain are shown in table 1.

| Table (1) Physical properties of Waste Glass, Porcelain Powder and Epoxy. |
|---------------------------------------------------------------|

\(^1\) The epoxy type used here is a commercial brand, EUXIT 50, supplied by SWISSCHEM, and the chemical composition cannot thus be identified as it is part of the intellectual property of the company and not available for public consideration.
### Material Specific Gravity Density (Kg/m³)

| Material   | Specific Gravity | Density (Kg/m³) |
|------------|------------------|-----------------|
| WG         | 2.6              | 2600            |
| Porcelain  | 2.4              | 2400            |
| Epoxy      | 1.05             | 1050            |

#### 2.2. Experimental Work

Composites of the epoxy were synthesized with filler (WG, porcelain powder) particles with 90-micron particle sizes as reinforcement. Different mixes of polymer composite were prepared to study their properties. The first set includes WG at 10, 20, 30, and 40%, while the other set included porcelain powder at 10, 20, 30, and 40%.

The filler was added to the resin and mixed with the resin for 2 to 5 minutes at 27 °C until a homogenous mixture was achieved. Then, then the amine hardener was added to the final mix. The final mixing process continued for a further 2 minutes at ambient temperature.

#### 2.3. Tests

The coating adhesion properties were measured via the obtained force and the type of failure occurring during the process. Porcelain and glass were chosen as additives due to their common high hardness. Adding these types of waste materials to epoxy increases the hardness of the resulting particulate composites. This was confirmed through a hardness test as described. The resulting composites with the addition of porcelain first and glass second were submitted to these hardness tests, revealing better readings for hardness than non-reinforced epoxy. To test the adhesion properties of the epoxy, a pull-off test was also carried out, offering a suitable indication for adhesion procedures.

#### 2.3.1. Density Test

The density of the particulate composites in kg/m³ were determined according to the standard procedure (ASTM C138) [5] of weighing the specimens and dividing these values (mass in kilograms) by the volumes of the specimens. Table 2 shows the results.

**Table (2)** Density of particulate composite specimens for glass and porcelain.

| Filler % | Glass  | Porcelain |
|----------|--------|-----------|
| 10       | 1120   | 1095      |
| 20       | 1244   | 1164      |
| 30       | 1290   | 1180      |
| 40       | 1318   | 1227      |
| epoxy    |        | 1050      |

#### 2.3.2. Hardness Tests
Hardness tests of the polymer-composite samples were carried out through recording the ratio of three readings from each side of the 50×50×50 mm cube, with the exception of the top face which was exposed to the air. As shown in Figure 1, a portable digital durometer for Shore D hardness testing was used as per ASTM D2240 [6].

![Digital durometer for Shore D hardness testing](image1)

**Figure (1)** Digital durometer for Shore D hardness testing

2.3.3. **Pull-off adhesion test**

Adhesion testing has become a popular method of quantifying the strength of paints and coatings. One of the most popular methods for investigating adhesion strength is the quantitative pull-off test. A pull-off test compliant with ASTM D-4541 [7] is one of the most universal procedures used for quality coating checks; this involves gluing the dolly to the concrete surface (as show Figure 3), then pulling the dolly using a force perpendicular to the surface to remove the dolly and the coating from the substrate.

The method includes an evaluation procedure, known as adhesion, which tests the strength of the pull-off for coatings on solid substrates such as concrete, metal, and wood. The test checks whether the surface maintains intactness at a specific prescribed force, resulting in either a pass or fail; the durability of the highest perpendicular strength of a certain surface area before the detachment of plug material can also be ascertained. This test uses portable pull-off adhesion testers (Self Aligning Type III (hydraulic)) [7] as show in Figure 2 and Figure 6. A hydraulic piston and pin are used to apply a load through the dolly centre. The size of the piston bore diameter is set to ensure that the area of the bore is equal to the dolly area. Thus, the reaction of the dolly pressure and the bore pressure are recorded simultaneously, and both are directly transmitted to a pressure gage.

![Type III (Self-Alignment Tester)](image2)

**Figure (2)** Type III (Self-Alignment Tester) [7].

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2 Figures 2, 4, and 5 are photos of equipment as used by the researcher, though the photos themselves are stock internet images. Figure 6 shows the actual equipment used by the researcher in the National Centre for Laboratories and Construction Research of the Ministry of Construction and Housing.
Preparing the Test Surfaces:
1) Dirt, chalking, oil and other interfering materials were removed
2) Smooth or glossy surfaces were lightly roughened using fine grit paper or an abrasive pad.
3) Any residual dust was removed using a compatible solvent.

To prepare the loading fixtures, a dolly with 19 mm outside diameter, was used. A 3 mm inside diameter hydraulic piston and pin was used to apply load to the dolly, and a hose, pressure gage, threaded plunger and handle were also affixed. Loading fixtures were abraded as necessary to enhance the bond with the surface and fixtures were cleaned before use as show in Figure 4.

A Teflon-fluorocarbon plug was inserted into the dolly until the tip protruded from the surface of the dolly. Adhesive (particulate composite) was applied to the loading fixture, the surface, or both as necessary. The loading fixture was attached to the coated surface (concrete) and pushed downward without twisting. The plug was removed after holding the dolly in place for 10 seconds as shown in Figure 5.
After seven days, when the composite was cured, a pin was inserted through the centre hole of the stainless-steel loading fixture (dolly). A pull upwards was given to the quick connect and the locking ring was allowed to engage the outside flange of the dolly. The pressure was increased slowly by turning the handle clockwise until either the maximum stress or failure was reached.

Figure (6) Actual equipment for Type III (Self-Alignment Tester) used in the pull-off test.

3. Results and Discussion

After conducting the hardness test to ensure that the particulate composite had good adhesion to the concrete surface and acted as a good coating material, a pull-off adhesion test was conducted.

3.1. Density Test

The presence of polymer induces an increase in the density of the composite because of the higher densities of glass and porcelain powder compared to epoxy. The particulate composite density thus varies depending on the amount of fine filler (glass or porcelain), and on how much air was entrapped or purposely retained. Table (2) shows the results of density tests for all proportions of epoxy with glass and porcelain as reinforcing materials.
Figure (7) Comparison of density and filler percentage in epoxy.

Figure 7 represents a comparison between densities at various filler percentages; the following observations can be concluded from the figure:

- The composite with 40% glass as a reinforcing material has the highest density.
- The density of glass composite is higher than that of porcelain composite at all proportions.
- Adding filler to the epoxy increases the density of epoxy at all ratios.

3.2. Hardness Test

The hardness test is mainly used for evaluating the indentation of materials. Such methods involve indenting samples using a steel indenter with specified force and geometry. It can be observed from the results of hardness testing that an increase in glass or porcelain content in the composite leads to an increase in the hardness values. Figures 8 and 9 show the results of the hardness tests for all proportions.

(A)  
(B)  

Figure (8) The effect of density on hardness for a) porcelain composites, b) glass composites)
The results seen in the above figures suggest that the addition of waste glass and porcelain to epoxy enhances the hardness value, due to the glass and porcelain having greater hardness properties. Thus, increasing of the percentage of glass and porcelain results in an increase of the value of hardness of the composite; higher hardness values are thus recorded with higher percentages of additions up to the 40% level. As the hardness of porcelain is higher than that of glass, the composite reinforced with porcelain demonstrates even higher hardness than the glass reinforced composite, as shown in figure 9.

Hardness values increased for all samples strengthened by glass powder, likely due to increased crosslinking and stacking, which reduces the movement of polymer molecules and leads to increased resistance to scratching and cutting. The composites are also more resistance to plastic deformation; where the material hardness depends on the type of forces between atoms or molecules in the material, additional stronger linkages increase the hardness value [8].

3.3. Pull-off test
Failure in the pull-off test for adhesion can either be a result of concrete substrate failure, as in the current work, or a failure occurring between the layers of the particulate composite. Concrete substrate failure is due to the adhesive force of the composite being greater than the cohesive force of the concrete, while failure between the layers of a particulate composite is due to the inverse relationship. Failure, occurring in the concrete (Figures 10 and 11) [9] suggests that the bond between the layers of the composite and the concrete tensile strength are smaller than the concrete interface.

**Figure (9)** Comparison between the hardness of different proportions of polymer composites and epoxy without reinforcement.

**Figure (10)** Pull-off test
(a) Cohesive failure within substrate, within topcoat.

(b) Adhesive failure between substrate and coating material.

**Figure (11)** Type of failure in pull-off test.

(A)

(B)

**Figure (12)** Effect of density on pull-off strength for A) porcelain and B) glass composite
Figure (13) Comparison between the pull-off strengths of different percentage of polymer composites and epoxy without reinforcement.

Figure 12(b) indicates that the addition of glass powder at all percentage between 10 and 40% creates an adhesive force within the composite greater than the cohesive force of the concrete. The pull-off strength for the glass particles composite is better than that of epoxy without reinforcing material.

In terms of the addition of porcelain, Figure 12 (a) shows that at percentages of 10 and 20%, the pull-off strength of porcelain approaches that of epoxy, with only a slight enhancement. This is due to the fact that the dominant ingredient in the composite is the matrix (epoxy), which causes the fillers to have less effect.

Conversely, at percentages of 30 and 40%, the pull of strength of porcelain composites increases noticeably, reaching 15 MPa and 16 MPa. This is due to the reinforcing material, porcelain, becoming more dominant in the composite; homogenous distribution of this porcelain in epoxy develops strength more than glass, making it a better coating material.

The results in Figure 13 show that adding porcelain and glass particles at all ratios improves the adhesive forces. Figure 14 also shows that at all ratios for composites with porcelain and glass, failure occurred in the concrete substrate as the bonds between the composite layers and between the concrete interfaces were greater than the tensile strength of the concrete [10].

Figure (14) Dolly cohesive failure shown on the concrete substrate.
4- Conclusions

The following observations can be drawn from the test results:

1- The final results showed a general improvement in the adhesion properties of composites when adding both glass and porcelain; nevertheless, porcelain results were much better than those seen with glass as filler with epoxy. This result achieved the main objective of the current work in terms of developing a better low-cost coating material for concrete substrates to be used for construction applications.

2- The composite with porcelain particles at a ratio of 40% gave the best results for the pull-off test (16.12 MPa), and the failure took place in the concrete substrate due to a good interface between the epoxy and filler particles.

3- For glass composites, the highest pull-off strength (5.81 MPa) was also for a 40% percentage at room temperature.

4- The composite with porcelain particles at a ratio of 40% gave the best results in the hardness test (98.62). For glass composites, the best hardness result (97.92) was also at the 40% percentage.

5. References

[1] Sasaki I and Nishizaki I 2008 Study on the Durability of Surface Coating Materials for Concrete Structures Iwao 2 305 8516
[2] Fletcher J F and Barnes D J 2017 Pull-Off Adhesion Testing of Coatings—Improve Your Technique
[3] Musa B H 2014 Effect of glass powder on some mechanical properties of polymer matrix composite material J. Al-Nahrain Univ. 17 108–15
[4] Croll S G Siriprom C and Keil B D 2014 Pull-off adhesion test for coatings on large pipes: possible variations in failure location and mode Pipelines 2014: From Underground to the Forefront of Innovation and Sustainability pp 1319–33
[5] ASTM C138/C138M 2013 Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) ASTM Int.
[6] ASTM D2240 2015 Test Method for Rubber Property--Durometer Hardness (West Conshohocken, PA: ASTM International)
[7] ASTM D4541 2009 Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion ASTM Int.
[8] Bhatnagar M S 2004 A textbook of polymers (New Delhi: S. Chand & Company)
[9] Borgerson J 2011 Evaluation of externally bonded fiber-reinforced polymer systems to concrete structures 18–21
[10] Hester R L 2015 Improving the accuracy of adhesion testing results

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