QUALITY CONTROL OF POLYMERIC MORTAR FLOORING SURFACINGS - RESIN CONTENT AND AGGREGATE GRADING BY PYROLYSIS

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QUALITY CONTROL OF POLYMER MORTAR FLOORING SURFACINGS

- RESIN CONTENT AND AGGREGATE GRADING BY PYROLYSIS

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Abstract:

Polymer mortars of epoxy, polyester or polyurethane resins and quartz or jasper sands were cast into cylinders, cured, compression tested to failure and then pyrolysed at 550°C. The resin content of the original mix was determined within +0 and -3% of the original. Analyses of the sand indicated the original grading. Techniques and sources of errors are discussed.
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INTRODUCTION

Polymer mortar floor toppings are used to prevent corrosion, impact and abrasion damage to concrete in industrial situations. They are normally site-applied by trowelling as a layer 3-10 mm thick. The polymer mortar is mixed on site from three base raw materials: resin, hardener, and sands. The resins used are epoxies, polyesters or polyurethanes, and the sands employed are usually hard, tough natural sands (such as quartz).

Subsequent to laboratory investigations and field studies carried out on specialised flooring materials for the food industry, a number of polymer mortar flooring failures have been investigated. In every case, the investigation was severely hampered by the lack of a specification for material or laying techniques, and a major lack of site quality control. In contrast, Portland cement concrete, a common industrial flooring material, is normally strictly specified and controlled. Polymer mortar floors represent a surcharge of $20-$50/m² on the basic concrete floor cost and thus are deserving of tighter specification control.

The purpose of this paper is to describe two methods of quality control. The new application of pyrolysis enables analysis of resin and sand content, and sand grading. The conventional compressive strength test assists with site quality control of batching.

METHOD

An exhaustive search of the literature unearthed no techniques for the analysis of hardened polymer mortar. For a related material, glass-fibre reinforced plastic, two methods are available for the determination of the resin content of the hardened material. These entail pyrolysis of samples to constant weight at 550° or 625°C.
Pyrolysis time, temperature

A few initial trials on polyester mortar using a propane gas burner showed that a pyrolysis method appeared feasible. Following these, samples (62.5 x 25 x 25 mm) of commercially prepared polymer mortar, surplus to earlier corrosion trials, were used.

To determine the length of pyrolysis time required, seven differently formulated prisms representing the range of resin types (i.e. 3 epoxy, 2 polyester, 2 polyurethane) were pyrolysed at 550°C as follows: the prisms were weighed (in an open porcelain crucible 100 diam. x 20 mm high), and placed in a preheated muffle furnace. After 1 hour they were removed to a dessicator, cooled, and weighed. This latter step was repeated a further three times.

To determine the effect of pyrolysis temperature, a further six prisms (2 epoxy, 2 polyester, 2 polyurethane) were pyrolysed for three four hour periods; the first four hours at 550°C, and the last two four hour periods at 625°C.

Effect of pyrolysis on uncombined aggregates

A possible complicating factor in the analysis of aggregate from pyrolysed polymer mortar is that quartz, one of the common sands, has an inversion temperature at 573°C. To investigate the effect of pyrolysis on aggregate grading, eleven samples of commercial sands (9 quartz, 1 jasper, 1 greywacke) were pyrolysed at either 550°C for four hours or 625°C for four hours. To find out if the sand broke down the grading and hence fineness modulus, was determined before and after pyrolysis.

Effect of pyrolysis on combined aggregates

Effect of pyrolysis on resin content

Compressive strength

In the case of Portland cement concrete, 28-day compressive strength is regarded as a guide to quality. Compressive strength testing of polymer concretes has been routinely carried out elsewhere (e.g. Ohama), and may similarly serve for quality control.

Steel moulds, 60 mm high and 30 mm in diameter, were made to the tolerances in NZS 3112: Part 2. The method used to form, cure and test polymer mortar cylinders follows.
A silicone release agent was sprayed into the mould prior to use. The cylinders were filled in three lifts, each layer being compacted with a minimum of 30 blows of a 12 mm diameter brass rod, and the top plate secured. After overnight curing at 20 ± 1°C, the cylinders were demoulded and cured at 20 ± 1°C and 65 ± 5% R.H., for a further 13 days. The cylinders were measured (height, diameter), weighed and any faulty ends capped with dental plaster in accordance with NZS 3112\textsuperscript{16}. Compressive strength testing was carried out (using a subpress) to ASTM D 695\textsuperscript{17}. Testing was carried out with the cylinders loosely enclosed in a plastic bag so that the shattered pieces were retained, and could subsequently be analysed by pyrolysis at 550°C for four hours.

The following mixes were used (all resin and aggregate quantities are by weight; sands used were naturally graded and not dried in the laboratory unless otherwise indicated).

a) 10, 15, 17.5, 20, 30% polyester resin (methyl ethyl ketone peroxide catalyst, cobalt octoate accelerator) with quartz sand as received.

b) 10, 12.5, 15, 17.5, 20, 25, 30% epoxy resin (polyamide hardener) with gap-graded quartz sand.

c) 10, 14, 18, 22, 26, 30% polyurethane resin (two part) with quartz sand.

Variations in the number of different resin contents used (5 polyester, 7 epoxy, 6 polyurethane) represent attempts to optimise this. For each mix two operators made three cylinders, giving six in all. Compressive strength, sand content, resin content by difference (i.e. weight resin/weight resin + sand) were determined for each cylinder, as described above except that the subpress required by ASTM D 695 was not available and hence not used for the polyester or epoxy samples. For all polyester and epoxy mixes, the six lots of pyrolysed sand were combined and the sand gradings and fineness moduli determined.

**RESULTS AND DISCUSSION**

**Pyrolysis time, temperature**

The effect of pyrolysis time on weight loss is shown in Figure 1. In all but one case (epoxy sample 3), the weight loss was negligible after two hours. Epoxy sample 3 was a partially filled resin (additional sand is added on site), and consequently very resin-rich
compared to site-applied flooring. It therefore appears that very resin-rich material needs a longer pyrolysis time than two hours, for epoxy sample 3 three hours were sufficient. Three hours at 550°C should therefore be regarded as a minimum pyrolysis time unless proved otherwise for a particular mix.

Apart from the resin-rich materials, the polymer contents tested 'as used', i.e. Epoxy 1, Polyester 1 and 2, and polyurethane 1 had resin contents (Table 1) generally in accord with industrial formulations of 12-20%\(^{18}\). Two of them were verified with the manufacturers concerned.

Extended pyrolysis at the higher temperature (625°C as opposed to 550°C) resulted in negligible additional weight loss for all systems tested.

**Effect of pyrolysis on uncombined aggregates**

Sieve analyses and fineness moduli before and after pyrolysis of the ten uncombined aggregates were virtually identical (e.g. Figure 2), implying that the presence of the quartz inversion temperature at 573°C does not affect pyrolysis of aggregates at 550°C or 625°C.

**Effect of pyrolysis on combined aggregates**

For ten resin/aggregate mixes tested, the changes in fineness modulus both for uniform and well graded as well as gap-graded sands, varied from a maximum of 0.18 to typically 0.03 (fineness modulus values were in the range 1.7 to 3.8). More specifically, there was negligible change in the size of material in the maximum size range. Part of the variation in fineness modulus is due to natural sieving error. Examples of grading curves and fineness modulus values before and after pyrolysis are shown in Figure 3 (naturally graded aggregate) and Figure 4 (gap-graded aggregate). The pyrolysing of resin and sand together thus does not affect the sand grading.

**Effect of pyrolysis on resin content**

The resin content of the polymer concretes, as determined by pyrolysis, is plotted against the known resin content of the mixes in Figures 5a (polyester), 5b (epoxy) and 5c (polyurethane). In all cases a good linear correlation was obtained (correlation coefficients 0.99, 1.00, 1.00, respectively), although the plots were displaced below the 1:1 relationship expected.
The following were excluded as reasons for the difference:

a) An initial moisture content in the sands. Measurements (to NZS 3111) gave a moisture content of 1.44% for jasper sand, and a range of 0.04-0.20% for quartz sands.

b) A lack of drying of cylinders prior to pyrolysis. Cylinders cured for 10 hours at 50°C and then dried for 24 hours at 105 ± 5°C suffered only a 0.5% loss in weight.

c) Crucible weight loss after 1 hour at 550°C was negligible.

d) The loss of volatiles from the polymers during the mixing was negligible. (A polyester resin, hardener and the two combined were left in an open dish in the laboratory and weighed at periods of 0.25, 1 and 2 hours. The weight losses were for the resin 0.097, 0.15 and 0.19%; for the hardener, 0.04, 0.31 and 0.71%; for the mixed components 0.14, 0.2, 0.22%. At the end of 6 hours exposure the weight loss was still 0.22%).

e) Ash in the resin. Hardened polyester resin was pyrolysed for 4 hours at 550°C and the resulting ash content was 0.05%.

The only obvious source of error remaining is the possibility of an excess amount of resin remaining on the mixing vessel and mixing rod. (Resin and hardener were weighed into the mixing vessel, so there can be no loss of material on the containing vessels). If this is the case, a better correlation would be expected from field tests.

The standard deviations for resin content are also plotted in Figure 5. The values for 30% resin content are high and may be caused by the high resin content which allows the sand to sink to the bottom of a mix and causes variability in sampling, handling of the material, and making of the cylinders.

**Compressive strength tests**

Plots of compressive strength against resin content are shown in Figure 6, and are similar to those noted elsewhere. Even rejecting the 10% and 15% polyester resin results (because they were determined on a hand-load-controlled testing machine), the coefficients of variation derived from standard deviations in this Figure show more variation than desirable; in some cases up to 9%. The normally accepted value for concrete compressive strength under NZS 3109 is 5%.
Possible causes of the extra error are:

a) The practice of capping only those ends which were not perfect, i.e. had holes in them or were not square. An improved technique could be to lie cylinders on their side as done in concrete testing, to try and avoid air bubbles on the top bearing face.

b) The second operator raised his tamping from 35 to 50 blows per layer to get more compaction.

c) The lack of experience of the operators who were working with this material.

d) Stiffening of the material towards the end of its potlife, and consequent increase in difficulty of working.

e) The difficulty of working material of high (> 20%) or low (< 15%) resin content.

ASTM D 695 requires at least five specimens for determinations of compressive strengths of polymer mortars. Bearing the above comments on variability in mind, five should also be quite suitable for resin content and sand grading determinations.

Although not feasible for the polyester mortars (apart from ensuring a minimum grade), limited correlation between compressive strength and resin content exists for both the epoxy and polyurethane mortars. This would have some value in site control, although initial tests would be required to establish the relationship for the particular epoxy mortar system in use.

Compressive strength tests also provide an indirect measure that formulations of the polymer mortar mix have been carried out correctly. Low values could indicate incorrect hardener proportions, or moisture in the sand (e.g. Ohama\textsuperscript{15}).

There is a definite link between the compressive strength of Portland cement concrete and such properties as abrasion resistance\textsuperscript{21}. This relationship is also inferred in much of the technical literature relating to polymer mortar (e.g. Bares\textsuperscript{20}), but requires more investigation.

For the present, compressive strength tests are seen as providing additional information for quality control of polymer concrete flooring, and although desirable are not essential.
CONCLUSIONS

1. Pyrolysis of polymer mortar samples at 550°C provides an easy and accurate method of determining the original resin content, sand content, sand grading, and fineness modulus. Testing at 625°C shows virtually no change from results at 550°C.

2. By casting the samples for pyrolysis in the shape of cylinders for prior determination of compressive strength, additional information is gained on the effectiveness of mix batching and the dryness of materials. The compressive strength may also provide a guide to potential durability of the polymer mortar.

3. The methods appear quite suitable for quality control of site-applied polymer mortar flooring.

4. Further work in refining sampling and testing techniques should increase the accuracy of the results.

ACKNOWLEDGEMENT

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REFERENCES


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</tr>
<tr>
<td></td>
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TABLE 1: RESIN CONTENT OF COMMERCIAL POLYMER MORTARS BY PYROLYSIS
Figure 1: Effect of pyrolysis time on weight loss

![Graph showing the effect of pyrolysis time on weight loss. The graph plots the percentage of original weight remaining against pyrolysis time at 550°C (hours). Different samples are represented by different markers: $E_1$, $E_2$, $P_1$, $P_2$, $U_1$, $U_2$, $E_3$.]

Pyrolysis time at 550°C (hours)

$E_{1,2,3} = $ epoxy mortar samples 1, 2, 3

$P_{1,2} = $ polyester mortar samples 1, 2

$U_{1,2} = $ polyurethane mortar samples 1, 2

Figure 2: Effect of pyrolysis on uncombined sand

![Graph showing the effect of pyrolysis on uncombined sand. The graph plots the cumulative percentage passing sieve against sieve opening size ($\mu$m). A dashed line represents before pyrolysis and a solid line represents after pyrolysis. The fineness modulus before pyrolysis is 3.28, after pyrolysis is 3.31.]

累计通过筛网百分比

筛网开口尺寸 ($\mu$m)

粗细度模数：

- 前未加热：3.28
- 后加热：3.31
Figure 3: Effect of pyrolysis on combined "as-received" sand

Polyester resin

fineness modulus before pyrolysis = 3.26, after = 3.25

Figure 4: Effect of pyrolysis on "gap-graded" sand

fineness modulus before pyrolysis = 3.27, after = 3.09
Figure 5: Actual and determined resin contents

- a) polyester mortar
- b) epoxy mortar
- c) polyurethane mortar

% resin in original mix vs. % resin determined by pyrolysis
14 day compressive strength, MPa

% resile content by weight

Polyurethane

Epoxy

Polyester

Figure 6: Resilience content versus compressive strength

Standard deviation
The Building Research Association of New Zealand is an industry-backed, independent research and testing organisation set up to acquire, apply and distribute knowledge about building which will benefit the industry and through it the community at large.