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QUALITY CONTROL OF POLYMER 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

Conference Paper 3

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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.

QUALITY CONTROL OF POLYMER MORTAR FLOORING SURFACINGS -

RESIN CONTENT AND AGGREGATE GRADING BY PYROLYSIS

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 thick¹. 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 sands¹ (such as quartz).

Subsequent to laboratory investigations²⁻⁵ and field studies^{1,6} 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 controlled⁷. Polymer mortar floors represent a surcharge of \$20-\$50/m² on the basic concrete floor cost⁸ 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^{9,10}. These entail pyrolysis of samples to constant weight at 550^o or 625^oC.

Pyrolysis time, temperature

A few initial trials¹¹ 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 trials², 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°C¹². 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 grading¹³ 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 quality¹⁴. Compressive strength testing of polymer concretes has been routinely carried out elsewhere (e.g. Ohama¹⁵), 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 2¹⁶. 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 $20 \pm 1^{\circ}\text{C}$, the cylinders were demoulded and cured at $20 \pm 1^{\circ}\text{C}$ and $65 \pm 5\%$ 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 3112¹⁶. Compressive strength testing was carried out (using a subpress) to ASTM D 695¹⁷. 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 3111¹³) 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^{19,20}. 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 3109²² 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. Ohama¹⁵).

There is a definite link between the compressive strength of Portland cement concrete and such properties as abrasion resistance²¹. This relationship is also inferred in much of the technical literature relating to polymer mortar (e.g. Bares²⁰), 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.

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REFERENCES

1. Sharman, W.R. 1983. Food factory floors: A guide to design, materials and construction. Building Research Association of New Zealand, Technical Paper P36. Judgeford
2. Sharman, W.R. and Cordner, R.F. 1978. Corrosion resistance of freezing works flooring materials. Building Research Association of New Zealand, Research Report R27. Wellington

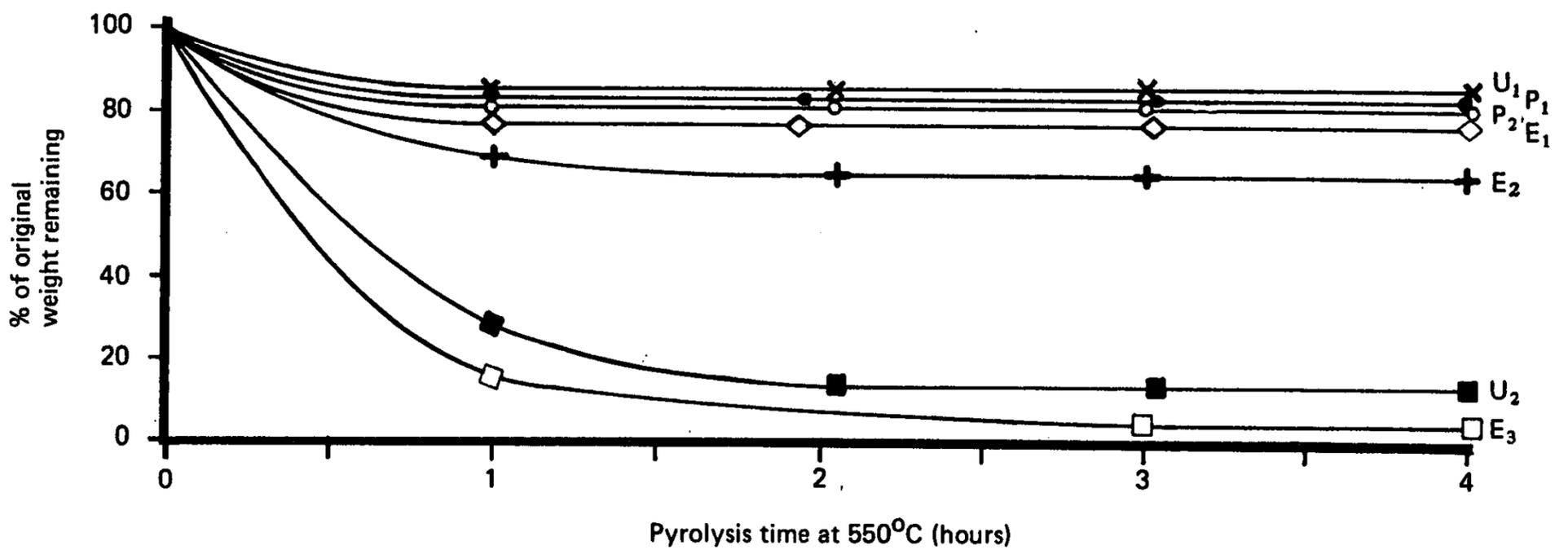
3. Sharman, W.R. 1978. Slip resistance of freezing works flooring materials. Building Research Association of New Zealand, Research Report R29. Wellington,
4. Sharman, W.R. and Cordner, R.F. 1979. Impact resistance of freezing works flooring materials. Building Research Association of New Zealand, Research Report R30. Judgeford
5. Sharman, W.R. 1982. Abrasion resistance of freezing works flooring materials. Building Research Association of New Zealand, Research Report R36. Judgeford
6. Sharman, W.R. 1976. An interim report on freezing works floors. Building Research Association of New Zealand, Technical Paper P12. Wellington
7. Perkins, P.H. 1973. Floors, construction and finishes. London, Cement and Concrete Association
8. Sharman, W.R. 1982. A directory of manufacturers and suppliers of specialised industrial flooring materials. Building Research Association of New Zealand, Technical Paper P15 (Third edition). Judgeford
9. British Standards Institution, 1977. British standard methods of testing plastics.
10. Glass reinforced plastics. Method 1002. Determination of loss on ignition BS 2782: Part 10: Method 1002
10. British Standards Institution, 1978. British standard methods of testing plastics.
10. Glass reinforced plastics. Method 1006. Determination of volatile matter and resin content of synthetic resin-impregnated textile glass fabric. BS 2782: Part 10: Method 1006
11. Sharman, W.R. 1980. Unpublished results
12. Lea, F.M. 1970. The chemistry of cement and concrete. London, Arnold. p.657
13. Standards Association of New Zealand. 1980. Specification for methods of test for water and aggregate for concrete. NZS 3111
14. Illston, J.M.; Dinwoodie, J.M. and Smith, A.A. Concrete, timber and metals: the nature and behaviour of structural materials. New York, Van Nostrand Reinhold. p 391

15. Ohama, Y. 1973. Mix proportions and properties of polyester resin concretes. American Concrete Institute Symposium Polymers in Concrete, SP-40: 283-294
16. Standards Association of New Zealand, 1980. Specification for methods of test for concrete. Part 2. Tests relating to the determination of strength of concrete. NZS 3112: Part 2
17. American Society for Testing and Materials. 1977. Standard test method for compressive properties of rigid plastics. D 695
18. Lilley, A.A. 1970. Experience in the U.K. in the application of thermosetting resin mortars for the surfacing and repair of concrete floors and roads. RILEM Symposium: Synthetic resins in building construction Vol. 2: 419-430
19. Inoue, S. 1976. Durabilities of cold setting epoxy resin mortars. Proceedings of the first international congress on polymer concretes: 191-194 (Construction Press, Hornby, Lancs)
20. Bares, R.A. 1978. Causes of polyester resin-filler surfacing systems failures. Second international congress on polymers in concrete: 311-330 (University of Texas at Austin, Austin)
21. Neville, A.M. 1973. Properties of concrete. London, Pitman (Second Edition)
22. Standards Association of New Zealand. 1980. Concrete construction. NZS 3109

TABLE 1 : RESIN CONTENT OF COMMERCIAL POLYMER MORTARS BY PYROLYSIS

| Resin Type | Sample No. | Resin Content (by weight) |
|--------------|------------|---------------------------|
| Epoxy | 1 | 22.3 |
| | 2 | 37.2 |
| | 3 | 97.2 |
| Polyester | 1 | 17.5 |
| | 2 | 18.4 |
| Polyurethane | 1 | 16.1 |
| | 2 | 86.8 |

Figure 1 : Effect of pyrolysis time on weight loss

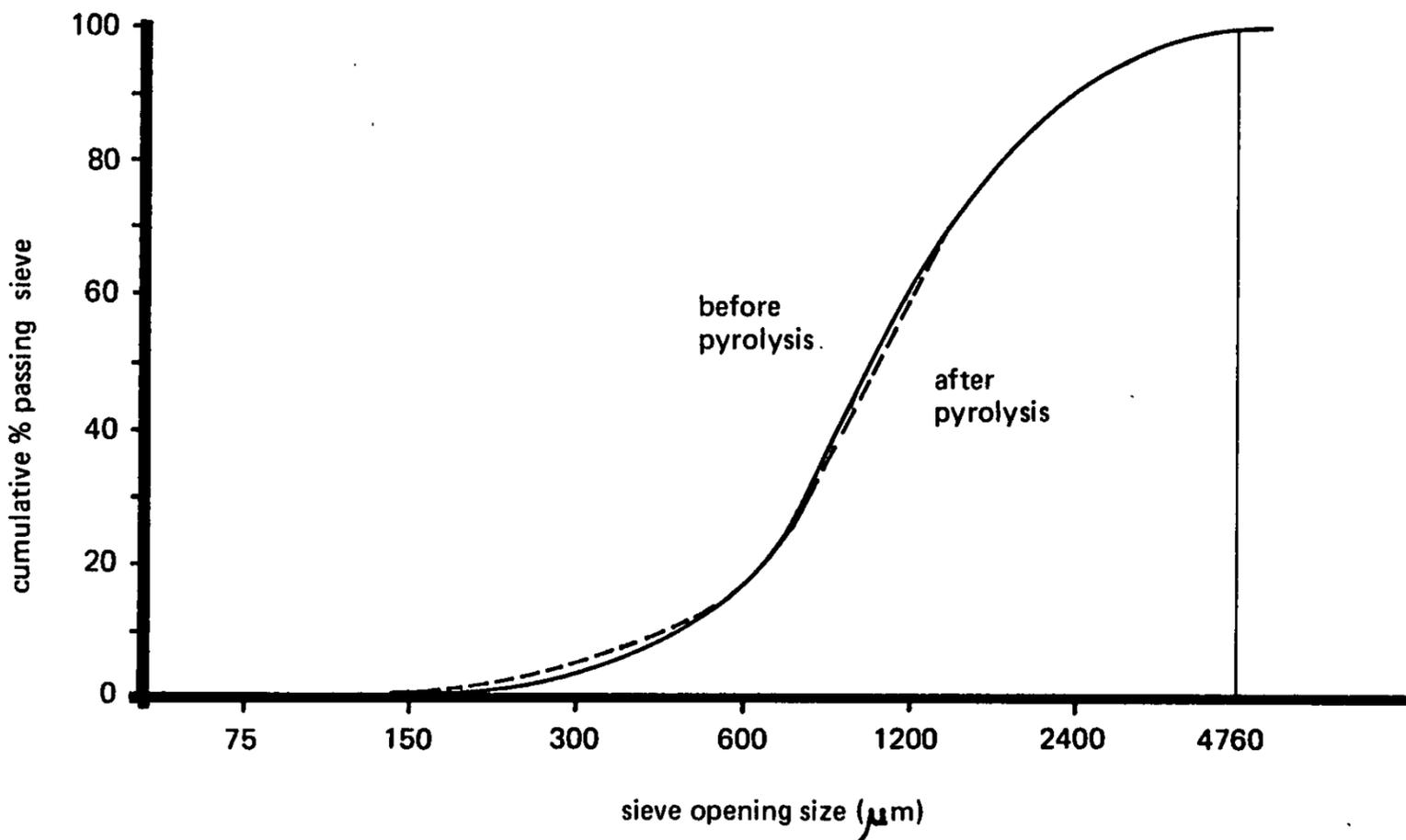


E_{1 2 3} = epoxy mortar samples 1, 2, 3

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

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

Figure 2 : Effect of pyrolysis on uncombined sand



fineness modulus before pyrolysis = 3.28, after = 3.31

Figure 3 : Effect of pyrolysis on combined "as-received" sand

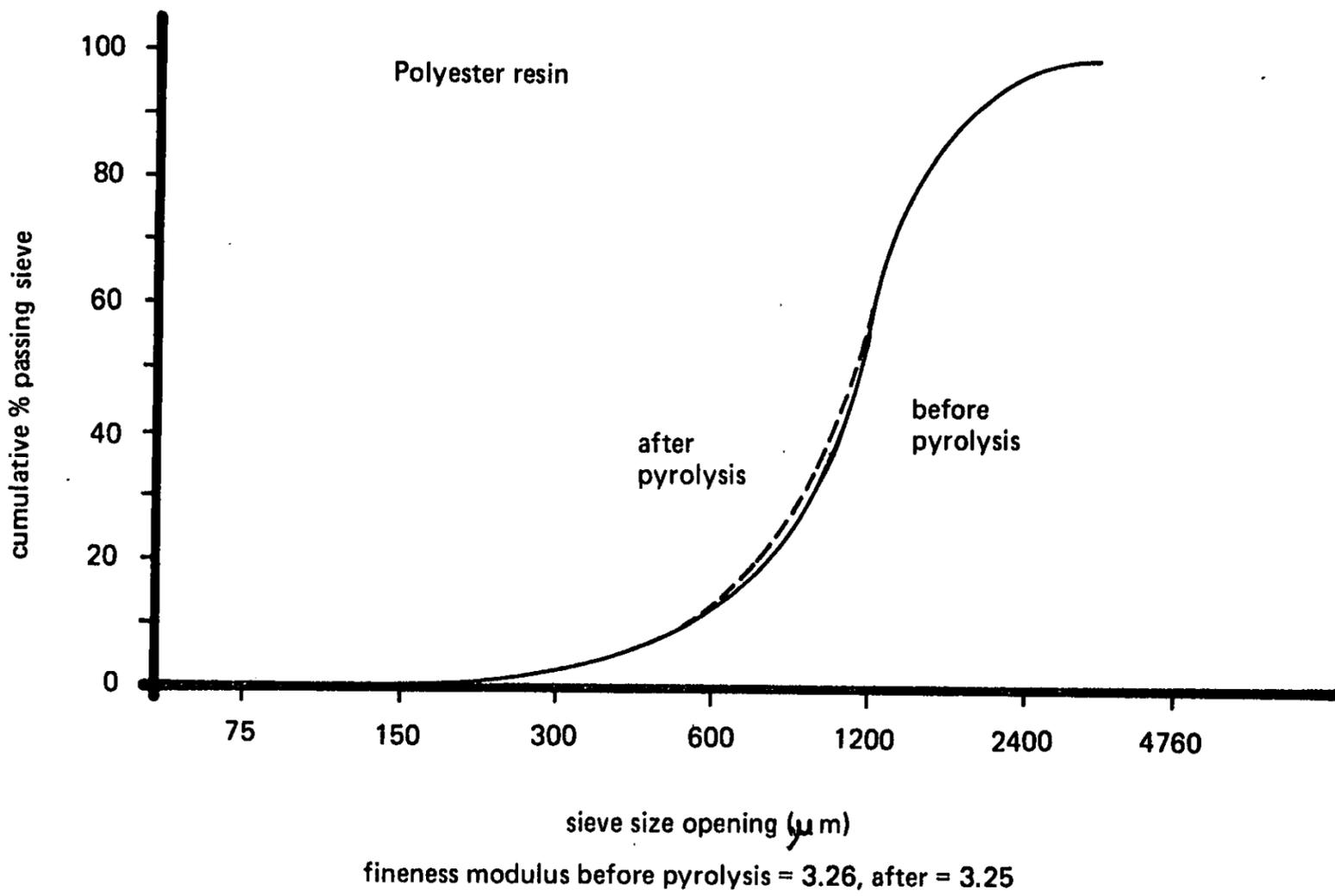


Figure 4 : Effect of pyrolysis on "gap-graded" sand

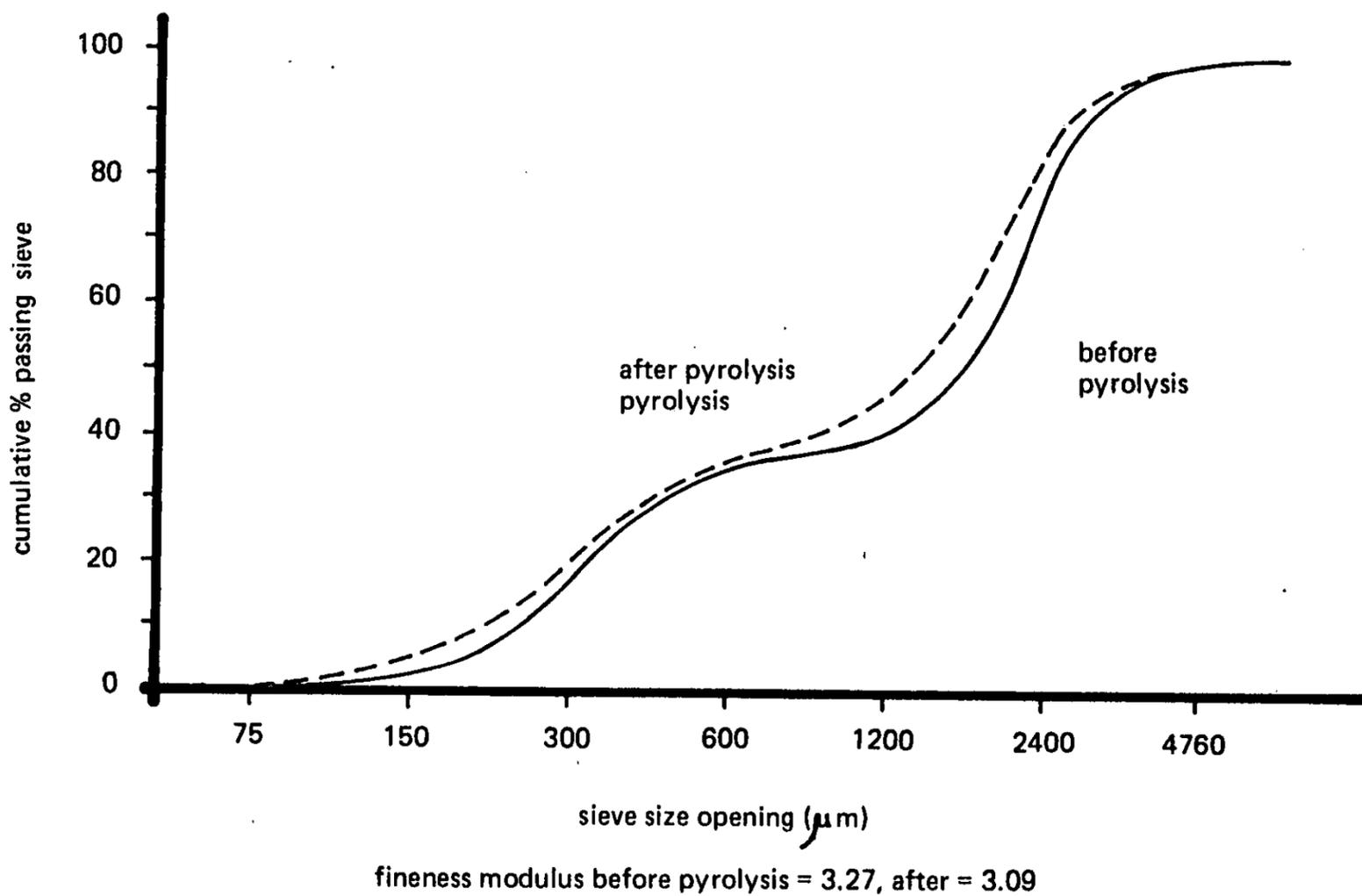


Figure 5 : Actual and determined resin contents

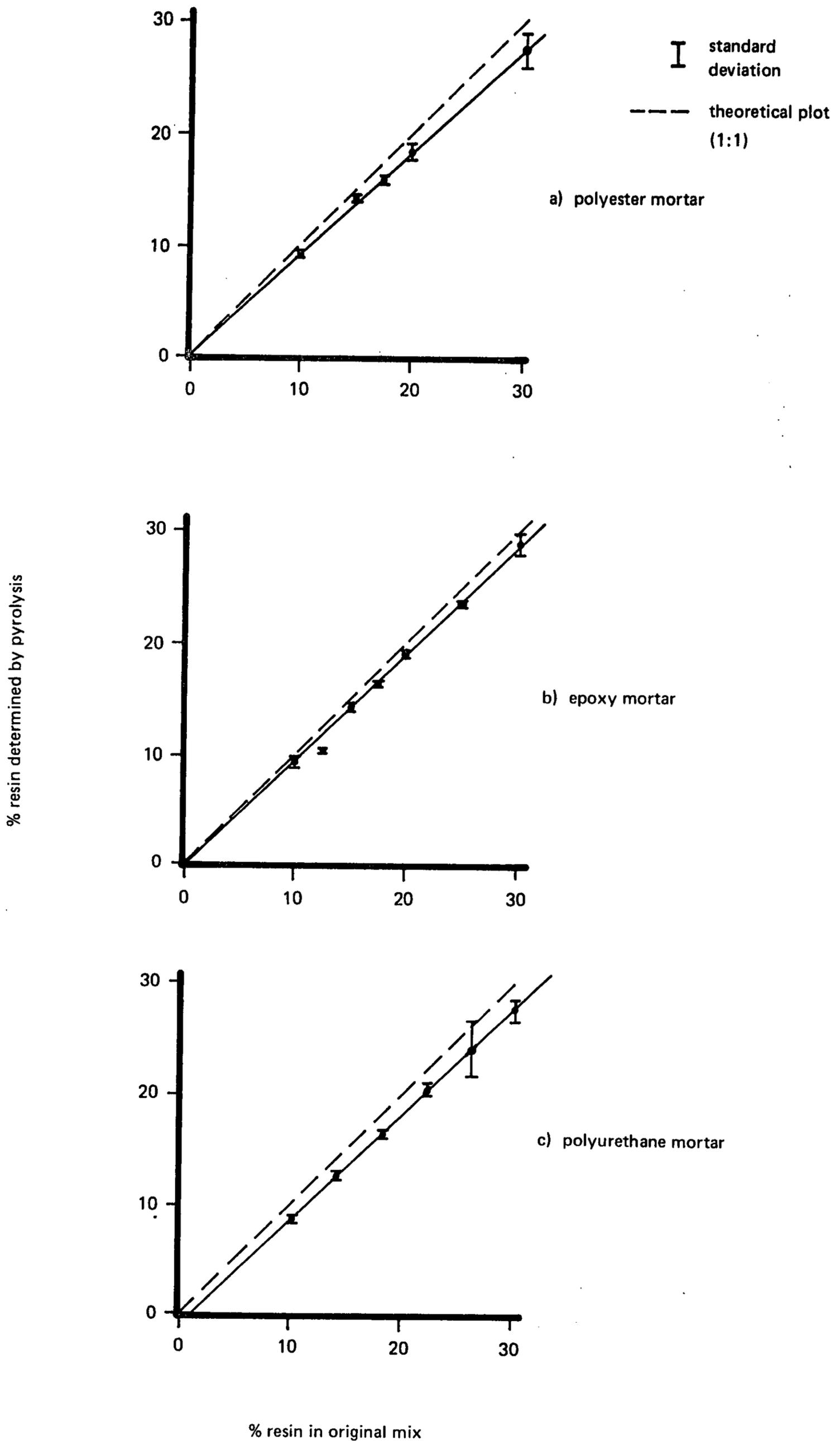
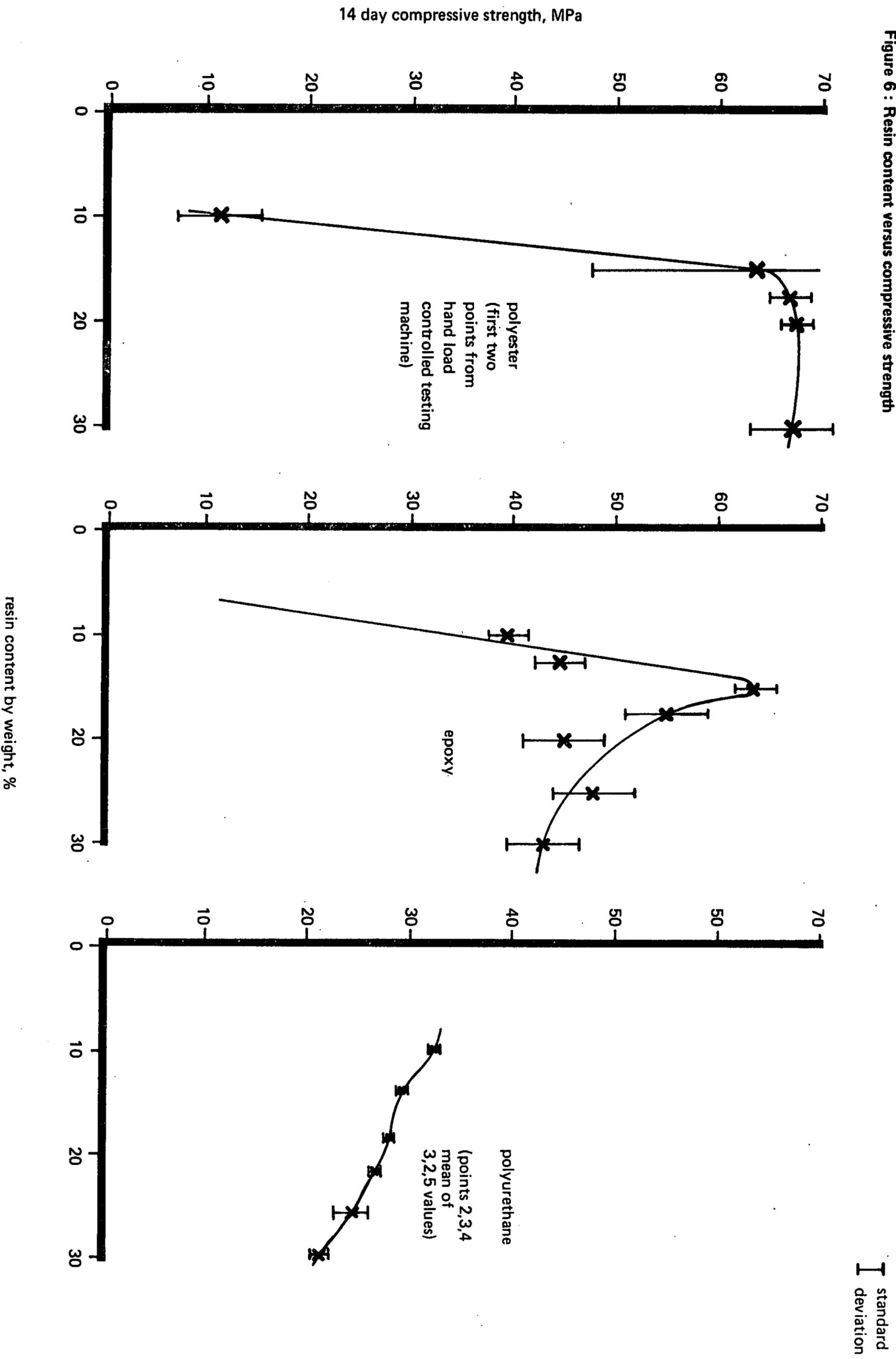


Figure 6 : Resin content versus compressive strength



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