*Proceedings of the 6th Asia-Pacific Structural Engineering and Construction Conference (APSEC 2006), 5 – 6 September 2006, Kuala Lumpur, Malaysia*

# EFFECT OF GGBFS AS FILLER IN POLYESTER GROUT ON STRENGTH DEVELOPMENT AND FLOWABILITY

**S. K. Lim[1], F. Zakaria[2],**
**M. W. Hussin[3], Z. Abd. Hamid[4], R. Muhamad[5]**
[1, 2, 3]Faculty of Civil Engineering, Universiti Teknologi Malaysia
[4, 5]Construction Industry Development Board (CIDB), Malaysia
Corresponding Author: S. K. Lim: siongkang@hotmail.com

ABSTRACT: Properties of polymer grout based on isopthalic unsaturated polyester resin (IUPR), methyl ethyl ketone peroxide (MEKP), river sand and ground granulated blast furnace slag (GGBFS) have been designed, tested and discussed in this paper. Slag was used as one of the design component to investigate its effect to polyester resin systems. Mechanical properties such as compressive and flexural strength of polyester grout under different curing conditions in tropical climate have been studied. It has been noted that GGBFS can be used as a micro-filler in polyester grout systems without weakening the mechanical properties of the resin systems. 10 percent to 30 percent of slag has been used to replace river sand, and the binder sand ratio is 1 to 1.5 by weight to maintain the flowability (pumpability) of polyester grout. At the higher level of slag, viscosity of resin system increased and caused the polymer grout gelatinous (unable to pump). The isophtalic polyester grout using slag as partial of filler provided compressive strength over 100MPa and the flexural strength is above 40MPa under all curing regimes. The polyester grout can be one of the good alternatives to replace epoxy grout as structural repair material as its superior properties closer to epoxy but it is cheaper than epoxy systems.

Key words: polyester, methyl ethyl ketone peroxide, ground granulated blast furnace slag, compressive strength, flexural strength

## 1. INTRODUCTION

Polymer or resin concrete serves as a unique concrete composite and becoming more popular in the construction industry in developed countries, particularly in the area of repair due to its easy application, quick setting characteristic, high mechanical strength, chemical resistance, wear resistance, controlled shrinkage and availability in differences viscosities [1]. Since the early 1960s the use of resin compositions in the construction industry has grown from very small beginnings to significant tonnages due to its bond strength that are considerably greater than the cohesive strength of concrete [2]. Polymer concrete (PC) and resin grout are produced by using dry aggregates which are mixed together with a thermosetting resin (binder) and curing agents/ hardeners till a homogeneous slurry and undergo polymerization (hardening). Thermoset resins are polymers that do not melt when heated, but decompose irreversibly at high temperatures. Thermoset resins posses a networked (cross-linked) structure, with the restrictive structure preventing melting behavior. Heating may form such a structure or via a chemical reaction Thermoset resins have excellent thermal stability and rigidity [3]. Various thermosetting resins have been used to prepare polymer concretes including epoxies, polyesters, phenol-formaldehyde (or phenolic) and furfural-acetone types. The composition of each polymer concrete composition is dictated by its application. The rate of development of mechanical strength and time saving is varying depend upon the resin system used, the ambient temperature and other factors.

In Malaysia, there is still limited knowledge of the properties and production of polymer concrete and resin grout. The present paper studies the design mix of resin grouts use in repairing industry that require compositions with higher fluidity (consistent flow). The binder used was isopthalic unsaturated polyester resin (IUPR) produced by local factory that cured with methyl ethyl ketone peroxide (MEKP). Fine graded dried river sand and slag (GGBFS) was used as filler in all these formulations. The various level of slag were added into the design mixes while in the same time maintained the fluidity of grout, the effect of slag on the mechanical properties (compressive and flexural) of IUPR grout under different curing regimes in tropical weather were investigated. It is essential to produce the design mix of local resin grout using IUPR that suitable for application in tropical climate. The mechanical properties of IUPR grout shall close to epoxy grout hence to replace the application of epoxy grout in repairing industry, which has higher cost than unsaturated polyester resin (UPR) grout.

## 2. EXPERIMENTAL PROGRAM

### 2.1 Materials

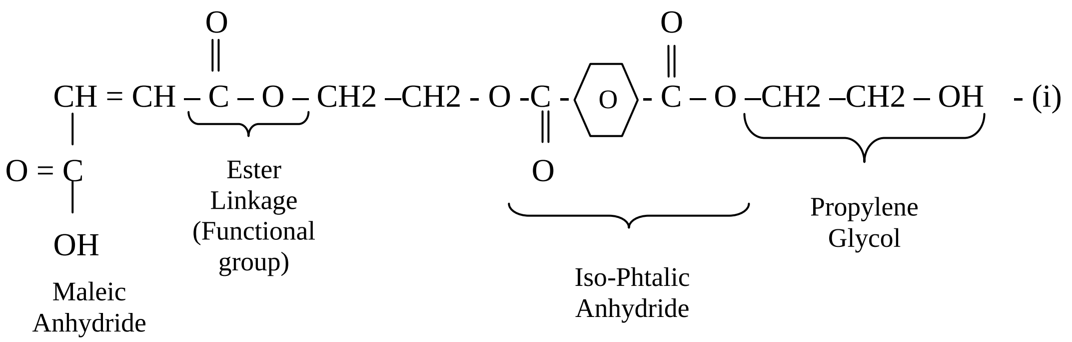
#### 2.1.1 Resin

Unsaturated polyester resin (UPR) was used in this study. According to Xia Cao and James Lee [3], UPR is one of the most common materials used in polymer concrete formulations because UPR can provide excellent mechanical and chemical properties, good chemical and weather resistance, and a lower cost compared with epoxy resin and is readily available in the market. Further advantages of UPR over other thermosetting resins are that they are easy to handle, can be pigmented, and can be easily filled and fiber reinforced in a liquid form.

Xia Cao and James Lee [4] state that UPR is macro-molecules with polyester backbone derived from the interaction of unsaturated acids or anhyhydrides and polyhydric alcohols. Solution of macro-molecules in reactive solvents like vinyl monomers (styrene, vinyl toluene etc.) are often called polyester resins. The cross-linking reaction between unsaturated polyester resin and vinyl monomers allows one polymer chain to connect with other polymer chains, and to produce a three dimensional network, which convert the resin system from a viscous liquid into hard, thermoset solid.

There are two types of unsaturated polyester resins, isophtalic and orthophtalic polyester. These two unsaturated polyester varieties (isophtalic and orthophtalic) show basic differences in their reagents. Charles [5] states that polyesters are the product of polycondensation reactions of dicarboxylic acids with dihydroxy alcohols. According to Gorninski et al. [6], Isophtalic polyester (iso-polyester) is produced from isophtalic acid while orthophtalic polyester is the result of a reaction with phtalic acid, which is different diacids. Because of the constraints involved in obtaining high molecular weight orthophtalic polyester, the properties of this material when used in isolation are inferior to those of isophtalic polyester. Gorninski et al. [6] claims that phtalic anhydride of orthophtalic polyester shows a strong tendency to regenerate from the ester medium of the phtalic acid (reversible reaction). This results in a larger amount of low molecular weight fractions, which are more susceptible to chemical attack. As the isophtalic acid does not form a cyclic anhydride in isophtalic resins, generation does not happen and high molecular weight polyesters are obtained with longer chains. As its carboxyl groups are more far apart (meta position), they do not interfere so much in the growth of the molecular chain of the polymer and the synthesis of long chains is possible, which results in increased mechanical strength in the end product.

This study investigates resin grout formulations prepared with P9728P isophtalic unsaturated polyester resin (IUPR) dissolved in styrene. The chemical structure and typical properties of IUPR is shown below:




*Equation (i) is linear isophtalic unsaturated polyester polymer chain*

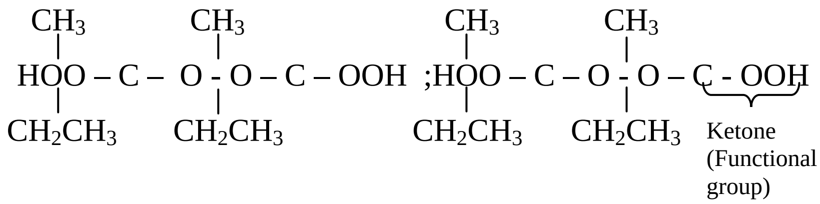
*Table 1: Typical properties of Iso-Unsaturated Polyester Resin P9728P (Ref. manufacturer's specifications)*

| | |
|---|---|
| Appearance | Pinkish Brown |
| Non-Volatile, % | 52 - 56 |
| Viscosity @ 25$^0$C, centipoises (Cp)<br>- Brookfield, #3/60 | 450 – 650<br>(Low viscosity) |
| Elongation (%) | 3.7 |
| Heat Distortion Temperature (HDT), $^0$C | 60 |
| Thixotropic Index @ 25$^0$C<br>- #3, 6 and 60 rpm | 1.5 – 2.8 |
| Gel time @ 25$^0$C, minute<br>- 1% MEKP | 24 - 30 |
| Acid Value, mgKOH/g<br>- Solid Resin | 25 |
| Specific Gravity | 1.1 |
| Volumetric Shrinkage, % | 9 |

### 2.1.2 Curing Agent for Polyester

According to BS 6319: Part 1 [7], hardener or curing agent is defined as a material that chemically combines with a synthetic resin to give a hardened product. Methyl ethyl ketone peroxide (MEKP) is widely used as curing agent of unsaturated polymer resin to mold products. MEKP is normally produced in the phlegmatizer (dimethyl phthalate, DMP) with acid as a catalyst. In the addition, the product with a concentration up to 10% active oxygen is neutralized, and then is brought to the desired concentration by further dilution with phthalate. According to Xinrui [8], MEKP is ordinarily a mixture of several isomers, all isomers contain the bivalent -O-O- linkage, and the molecules and their anions are powerful nucleophiles.

For this study, MEKP in dimethyl phthalate was used to cure the UPR. MEKP is clear and colourless liquid. It is organic peroxide. The chemical structure of MEKP is shown below:




*Equation (ii) is methyl ethyl ketone peroxide in dimethyl phthalate chemical structure*

#### 2.1.3 Filler

According to BS 6319: Part 1 [7], filler is defined as a solid material in powder, granular or fibrous form that is added to a synthetic resin system to reduce cost, exothermic and shrinkage and often increase hardness, abrasion resistance, heat distortion temperature (HDT) or other specifically selected property of a cured system. Fillers may also modify the flow properties of the system before cure.

Dry graded river sand was used as major filler in this study. The sand used is complied with ASTM C 778 – 91 [9]. According to Houlsby [10], the main purpose using fine sand to produce grout is to reduce cost by decreasing the usage of binder. The strength of grout will be reduced and viscosity will increase if the binder aggregate ratio is higher.

Ground granulated blast furnace slag (GGBFS) is used as powder filler (macro-filler) because it is widely available in Malaysia. Golding [11] state that powder fillers are added to increase viscosity, improve abrasion resistance and gap-filling properties, impart specific electrical or mechanical properties, or reduce cost and shrinkage. According to ACI 233R [12], GGBFS is a by-product of the steel industry. It is glassy granular material that is formed when molten blast furnace slag is rapidly chilled by contact of water (“granulated”), dried and ground to a fine powder. According to Pal, Mukherjee and Pathak [13], the specific gravity of the slag is approximately 2.90 with its bulk density varying in the range of 1200-1300kg/m3. The colour of GGBFS is normally whitish (off-white). The physical properties of GGBFS usually results in enhanced the hydraulic potential, longer time of set at temperature less than 85 degree Fahrenheit (29 degrees Celsius), higher 28-day strength, reduced bleeding, lower permeability, resistance to sulfate attack and mitigate alkali-silica reactivity (ASR), more durable and lighter colour. The GGBFS used for this study is obtained from YTL (Malaysia) Sdn. Bhd.

### 2.2 Test Methods

The compressive test was performed according to ASTM C 579 – 01 [14] and equivalent to BS 6319: Part 2 [15]. 50mm x 50mm x 50mm cube has been selected as the specimen size. Specimens were tested at a rate from 1.5 to 1.8kN/s according to ASTM C 579 – 01. Three cube test specimens were prepared for each period and curing condition and results are the mean of individual results.

The flexural test was carried out in accordance to RILEM PC 2 (1995) [16]. Prisms of size 160mm long x 40mm width x 40mm depth are used for this method which is otherwise a standard center point load flexural test. . Imposed the prisms with a center point loading system. Apply the load at the rate between 0.09 to 0.13 kN/s. Measure the position of the crack from the support.

### 2.3 Exposed Conditions

In this paper, five different exposed conditions have been carried out to determine its effect on the durability, compressive strength and flexural strength, resistance to chemical attack such as sulfate from muddy soil and chloride content in sea water. After 24 hours, the test specimens were demoulded and subjected to the condition as shown in Figs 1 to 6:

*Fig. 1: Air curing in laboratory. Average temperature At 30$^0$C with 65% relative humidity*

*Fig. 2: Tropical climate outside laboratory. Temperature range from 26$^0$C (raining) to 38$^0$C (hot) with humidity range from 25% (hot and dry) to 90% (wet)*

*Fig. 3: Continuous water curing at 25 – 26$^0$C*

*Fig. 4: Wet-dry cycles. Immersed in water per week and exposed to open air per week consider as one cycle*

*Fig. 5: Flow-ebb of seawater. Samples were Immersed in seawater*

*Fig. 6: Samples under seawater curing condition*

### 2.4 Polyester Grout Compositions.

The compositions used in the polyester grout formulations in this study are listed in Table 2. The design of the polyester grout was based on cost effective, pumpable, sufficient working life (more than 30 minutes) and targeted strength. GGBFS was added into the polymer matrix to reduce settlement of the fine sand, reduce volume shrinkage, enhanced the homogeneous of polymer matrix. 10 to 30 percent of GGBFS was used to replace the content of fine aggregate to study its effect to polymer matrix on strength development and durability while in the same time maintained the consistent flow of polyester grout.

*Table 2: Polyester grout compositions*

| Polymer matrix components | Composition | % of total mass |
|---|---|---|
| (a) Polyester<br>Resin | Unsaturated isophtalic polyester P9728P | NIL |
| Hardener/ starter | Methyl-ethyl-ketone peroxide (MEKP) | NIL |
| (b) Fine aggregates<br>Sand | Oven dried river sand, fine particle size based on ASTM C 778 | NIL |
| Powder-filler | Ground granulated blast furnace slag (GGBFS) | (0, 10, 20 and 30)[c]<br>(10 – 30)[d] |

Note:
[c] Percentage in relation to the sand mass
[d] Range (grams) for every 100g (total mass) of polyester grout

## 3. RESULTS AND DISCUSSION

The results for compressive strength and flexural strength of polymer samples prepared with isophtalic polyester and 10 to 30 percent GGBFS, which subjected to five curing regimes, are presented and discussed. The durability determination was based on the strength development of samples that exposed to different conditions compared with normal curing condition for resin grout (open air). The chemical resistant test was based on the durability (mass loss caused by corrosion) and strength of samples, which were exposed to effect of flow-ebb of seawater, attacked by chloride and sulfate content (3 to 5%) in seawater and muddy soil.

The following codes were used to present the results:

I = samples prepared with unsaturated isophtalic polyester.

Numbers (0, 10, 20, 30) = percentage of GGBFS used in relation to the mass of fine sand.

### 3.1 Flowability and Gel Time

It was found that when the GGBFS content increased in the grout mixes, the flowability of the grouts was decreased when tested with rotational viscometer. This is because the GGBFS seems like soluble in polymer matrix and make its shear rate higher. When the shear rate higher, the viscous of the grout mixes increased. The viscosity of resin grout is suggested to be around 2500 centipoises (low medium viscosity) or below tested with Brookfield viscometer using spindle 3, 60rpm to ease the pumping or injection works. It was found in this study that adding of 0 to 30 percent GGBFS into polyester resin matrix maintained the consistent flow of the grout and the viscosity of grout mixes were in the range of 1550 to 2500 centipoises at spindle 3, 60rpm, $30^0$C. The gel time of the resin mixture mainly control by the hardener and accelerator. The accelerator like cobalt is used to reduce the gel time of resin mixture, it is not suitable to use the accelerator in this study while the resin grout must has longer and sufficient working time for pumping. MEKP was added into the resin matrix to cure it. The percentage of hardener was determined by the objectives to have sufficient working time and maintain the strength required. It was also found that when adding more GGBFS, the increasing of viscosity of grout mixes was slightly shorten the gel time or working time of resin matrix. Table 3 shows the gel time of polyester grouts measured in accordance to ASTM D 2471[17]:

*Table 3: Gel time of polyester grout*

| **Polyester grout** | I-0 | I-10 | I-20 | I-30 |
|---|---|---|---|---|
| **Gel Time (minute)** | 33-37 | 31-34 | 30-33 | 30-33 |

### 3.2 Compressive Strength and Durability

The density of polyester grouts is in the range of 1760 to 1800kg/m3. Increase of slag content increased the weight of grout mixes. The curing conditions have no much effect of the density changes even exposed to tropical climate or immersed into water. The polyester matrix is very solid after fully cured and posses thixotropic (repelled water) characteristic. Figs 7 to 9 show the effect of environment (curing condition) and GGBFS concentration on compressive strength of isophtalic polyester at 7, 28 and 90 days.

The results summarized in Figs 7 to 9 show high compressive strength values (all above 75MPa). These results fulfill the requirement anticipated for polymer concrete or grout.

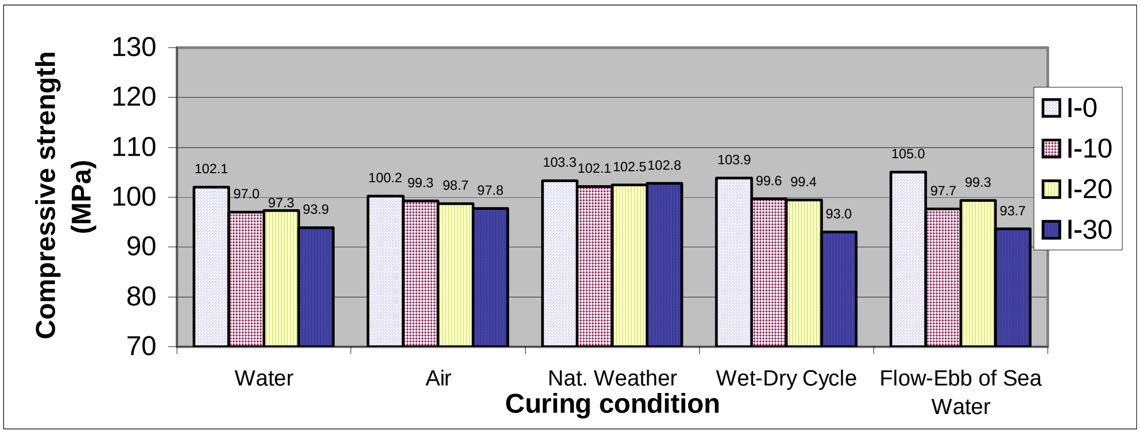



*Fig7. 7-days compressive strength of polyester grout*

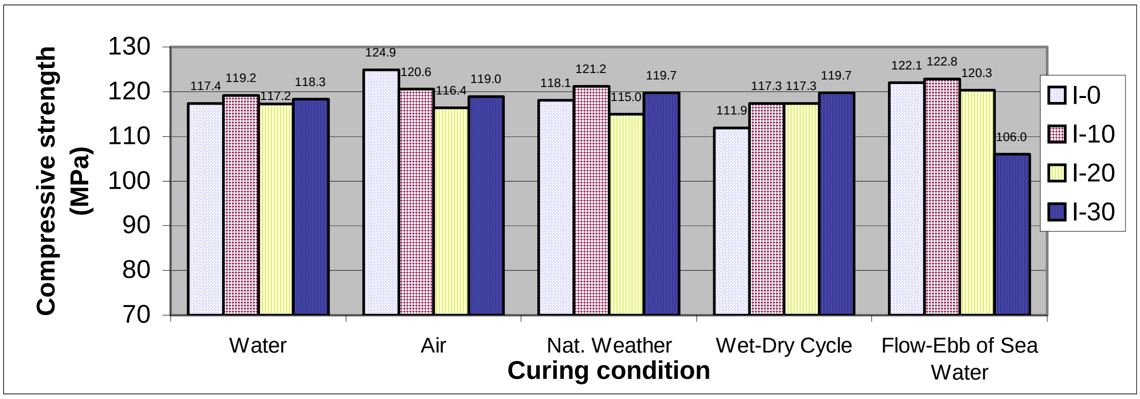



*Fig 8. 28-days compressive strength of polyester grout*

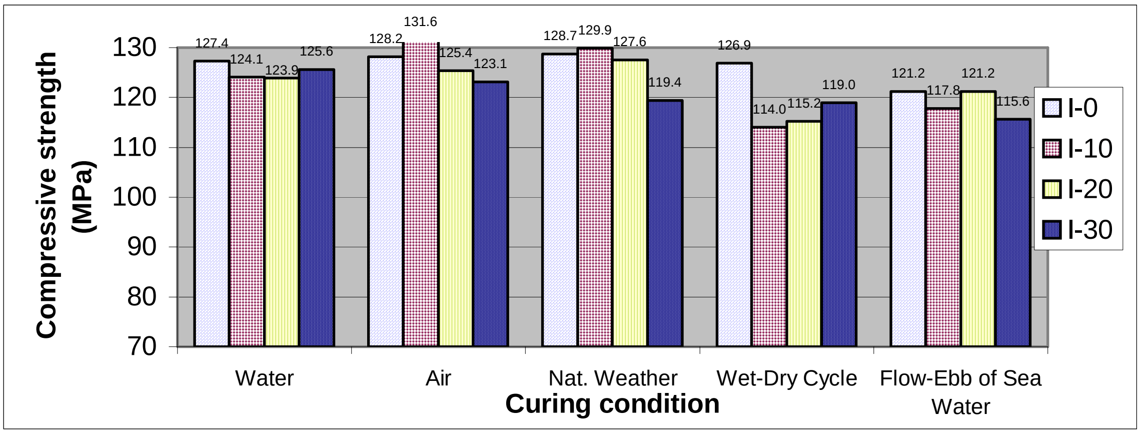



*Fig 9. 3-months compressive strength of polyester grout*

Figs 7 to 9 show values in range of 90MPa to 130MPa using isophtalic polyester concentration of 40 percent, fine graded sand and GGBFS (or not) as filler. It was found that changes in GGBFS concentration in range 0 to 30 percent have little effect in compressive strength development. Based on Fig. 7, the samples with GGBFS gained lesser strength compared to control samples (I-0) on day-7. It may caused by GGBFS has slower down the process of polymerization of resin matrix to bind the filler. The GGBFS added into the grout mixes make the mixes more homogeneous, increased density, reduced volume shrinkage during polymerization and enhanced elasticity.

The curing conditions have little effect to the compressive strength development. Samples under tropical climate have almost similar results with air-cured samples (reference) at 28 days and 3 months. When the weather was hot (no rain), the network forming process of samples was accelerated by heating and gained higher compressive strength compared with reference samples within 7 days. Barbara Stuart [3] stated that thermoset resin formed its long chain cross-linked structure (become solid) by heating or via a chemical reaction. Based on results indicated in Fig. 9, all seawater samples only slightly decreased its compressive strength. Based on Fig. 7, the 7-days samples immersed into water (even sea water) or via wet-dry cycle normally have lesser strength compared with air-cured samples. This may caused by the low temperature and wet condition slower the polymerization process of samples, water inside samples also reduced the bonding of polymer matrix to filler. The results from seawater samples compared with air curing samples proved the isophtalic polyester mixes could resist to chloride and sulfate attacks. From the results showed in Figs 7 to 9, the samples normally achieved its optimum strength at 28 days and maintain the strength afterwards. The samples achieved its compressive strength around 85 percent of optimum strength at day-7. There were no weight loss nor deteriorate of polyester grout samples exposed to environmental changes even flow-ebb of seawater. The isophtalic polyester compounds are durable under all studied environmental conditions.

### 3.3 Flexural Strength

Fig. 10 shows the effect of curing condition and GGBFS concentration on flexural strength at day-90. The values obtained from the current investigation, as showed in Fig 10., indicated that these polyester compositions reached excellent flexural strength and achieved one third of compressive strength. The flexural strength of all polyester prisms reached 50 to 55MPa at 3 months period under five study conditions. The values obtained in the present study are very high if compared to cement-based grout. In the latter study, the author used 50 percent GGBFS to replace ordinary Portland cement and 0.58 w/b ratio to produce slag cement based grout. The 6-months mean values for flexural strength of slag-cement based grout reached 9.40MPa (water-cured) and 5.71MPa (tropical weather) for a compressive strength level of 60MPa and 40MPa. From the results obtained, we can notice that the flexural strength of isophtalic polyester grouts reached 40 to 45 percent of the value of compressive strength. However, flexural strength for cement-based grout only achieved 10 to 15 percent of the value for compressive strength.

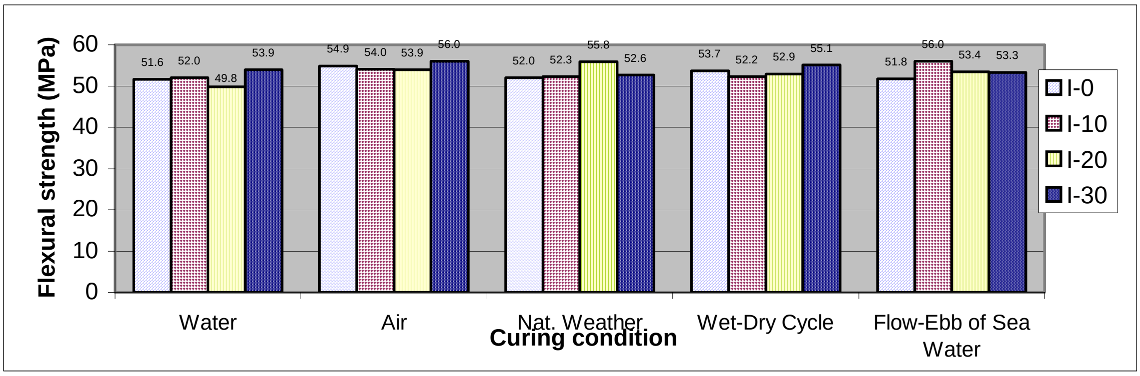



*Fig 10. 3-months flexural strength of polyester grout*

## 4. CONCLUSION

Based on the-above investigations, we can conclude that replacement of 10 to 30 percent GGBFS as part of filler developed compressive strength more than 90MPa after 7 days under all study conditions. The replacement of slag in the range can maintain the consistent flow of resin grout for pumping purpose. When slag content increase, the strength development will get slower in short-term, this caused the slag based polyester grouts have lower compressive strength at day-7. The compressive strength of all polyester grouts almost the same at day-28 and 3-months. The exposure of the specimens to the Malaysia tropical weather has no effect to the strength development of isophtalic UPR grout. The polyester grout can maintain its strength and durable under tropical climate. The polyester grout also has good resistance to seawater effect. The flexural strength of polyester increased when GGBFS was used in the grout mixes. The micro-particle size of GGBFS enhanced the elasticity and homogeneous of casting samples. It was found that the slag based polyester grout gained higher compressive strength in long-term period compared to control mixes. The interface bond between GGBFS and polymer matrix took longer time than polymer-sand interface bond, but this will make the samples reduced its voids and become more solid.

## 5. AKNOWLEDGEMENTS

This research was carried out in the Structure and Material Laboratory of Faculty of Civil Engineering, Universiti Teknologi Malaysia. Financial support from the Construction Industry Development Board of Malaysia (CIDB) is greatly appreciated.

## 6. REFERENCES

1. Moetaz M. El-Hawary and Hisham Abdel-Fattah (2000). Temperature effect on the Mechanical Behavior of Resin Concrete. Construction and Building Materials. 14: 317-323.

2. Shaw J. D. N. (1985). Resins in Construction. Cement Composites and Lightweight Concrete. Proceedings of the Seminar Resins in Construction, London. 7(4): 217-223.

3. Barbara H. Stuart (2005). Polymer Analysis. Sydney. John Wiley & Sons, Ltd.

4. Xia Cao and L. James Lee (2002). Control of Shrinkage and residual styrene of unsaturated polyester resins cured at low temperatures: I. Effect of curing agents. Polymer. 44(6): 1359.

5. Charles E. Carraher, Jr. (2003). Polymer Chemistry. Sixth Edition. New York. Basel: Marcel Dekker, Inc.

6. Gorninski J. P., Dal Molin D. C. and Kazmierczak C. S. (2005). Comparative Assessment of Isophtalic and Orthophtalic Polyester Polymer Concrete: Different Costs, Similar Mechanical Properties and Durability. Construction and Building Materials. 14: 1-10.

7. British Standard (1983). Method for Preparation of Test Specimens. Testing of Resin Compositions for Use in Construction (BS 6319: Part 1)

8. Xinrui Li, Hiroshi Koseki, Yusaku Iwata and Yun-Soo Mok (2004). Decomposition of Methyl Ethyl Ketone Peroxide and Mixtures with Sulfuric Acid. Loss Prevention in The Process Industry. 17: 23-28.

9. American Society for Testing and Materials (1991). Standard Specification for Standard Sand (ASTM C 778 - 91).

10. Houlsby A. C. (1990). Construction and Design of Cement Grouting - A Guide to Grouting in Rock Foundations. Canada: John Wiley & Sons, Inc.

11. Goulding T. M. (1994). “Epoxy Resin Adhesives.” Handbook of Adhesive Technology, New York, Marcel Dekker Inc., pp. 1-13, 531-546.

12. American Concrete Institute (1995). Ground Granulated Blast-Furnace Slag as a Cementitious Constituent in Concrete (ACI 233R-95).

13. Pal S. C., Mukherjee A. and Pathak S. R. (2003). Investigation of Hydraulic Activity of Ground Granulated Blast Furnace Slag in Concrete. Cement and Concrete Research 33, London: Elsevier Science Ltd., 1481-1486.

14. American Society for Testing and Materials (2001). Standard Test Method for Compressive Strength of Chemical-Resistant Mortars, Grouts, Monolithic Surfacings, and Polymer Concretes (ASTM C 579 - 01).

15. British Standard (1983). Method for Measurement of Compressive Strength. Testing of Resin Compositions for Use in Construction (BS 6319: Part 2)

16. RILEM Technical Committee (1995). Method of Making Polymer Concrete and Mortar Specimens. Symp. On Properties and Test Methods for Concrete Polymer Composites. Proc. Oostende-Belgium. TC-113. 129-132 (RILEM-PC2)

17. American Society for Testing and Materials (1988). Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins (ASTM D 2471 - 88).