

Studies on the Preparation of Mortar-Plastic Composite

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Abstract

The preparation method of the mortar-plastic composites(M P C) were studied with styrene, methyl methacrylate and vinyl acetate as monomers. Radiation, thermal-catalytic and radiation-catalytic methods were used as curing methods. Almost all of the above monomers and methods were possible to use for preparing M P C. Although thermal-catalytic method was excellent to get M P C in a short time, the tensile strength of the product was less than those obtained by radiation method. It was possible to prepare the M P C which included up to about 10% plastics and was strengthened about ten times on the properties of acid resistances and tensile strength comparing with the control. The improvement of the properties is much superior to concrete-plastic composite(C P C).

요 약

단량체로서 Styrene, Methyl methacrylate 및 Vinyl acetate 를 사용한 몰탈-플라스틱 복합체 제법에 관하여 검토하였다. 경화법으로서는 방사선, 열-촉매 및 방사선-촉매 방법을 사용하였다. 상기한 단량체 및 중합방법은 모두 복합체 제조에 사용이 가능하였다. 열-촉매 방법이 중합에 가장 적은 시간을 요하였으나 이 방법에 의한 제품의 인장강도는 방사선에 의한 제품에 비하여 열등하였다. 플라스틱을 약 10%까지 함유시킬 수 있었으며 이렇게 하므로써 처리치 않은 몰탈에 비해 약 10배의 내산성 및 인장강도 등의 성질향상이 가능하였다. 이 성질의 향상은 콘크리트-플라스틱 복합체에 있어서 보다 훨씬 컸었다.

1. Introduction

Since the first study¹⁾ on the preparation of concrete-plastic composite(C P C) with the

similar method used for wood-plastic combination²⁻⁵⁾ was reported in 1968, extensive studies in this field has been continued⁶⁻⁸⁾. The results of the studies are as follows; (i) impregnation of concrete with monomer and

followed curing the monomer by radiation or thermal-catalytic process is useful for the preparation of the composite, (ii) marked improvement in physical and chemical properties of the concrete is obtainable by the treatment. In this study, the authors tried to use mortar in which no pebble is contained as a raw material, instead of concrete used by above research groups⁶⁻⁸⁾. To investigate if mortar as good as concrete can be made to useful composite of the plastics, the processes of the preparation have been examined with the properties of the products.

2. Experimental

1) Materials and Instruments

a) Mortar specimens;

The mixture made of cement, sand (above 20 mesh) and water in ratio of 1:3:0.5 is cast to ϕ 2.5×5 cm cylindrical mortar. This mortar is left in the vacuum desiccator with the some water on the bottom for 24 hours, and it is cured in the steam bath for 24 hours. Drying is carried out in drying oven at about 130°C.

b) Styrene;

The technical grade monomer is mixed with 15% sodium hydroxide aq. solution in 4:1 volume ratio and vigorously agitated. After removing the aqueous layer, the monomer is washed twice with distilled water, dried with calcium chloride, and distilled under reduced pressure.

c) Methyl methacrylate(M M A);

The monomer is washed with saturated aq. solution of hydrogen sulfite, and 5% sodium hydroxide aq. solution using 10:1 volume ratio to remove the stabilizer. The purification procedure is similar to those of styrene beside the inhibitor removing process.

d) Vinyl acetate(V A):

To 5 parts of the monomer 1 part of 10% sodium acetate aq. solution is vigorously mixed and the aqueous layer is separated to remove the stabilizer. Then it is purified by distillation.

e) Catalyst;

Benzoyl peroxide(B P O) made in Kawaguchi Yakuhin Co. in Japan is used as catalyst without any further purification.

Radiation source;

f) The Co-60 gamma source, around 500 Ci, is used after intensive measurement of dose rate by chemical dosimetry.

2) Preparation of M P C

a) Impregnation of monomers;

To the styrene monomer, 0.2% of hydroquinone is added to prevent probable polymerization during impregnation experiment. To measure the amount impregnated in different methods of impregnation and different duration of dipping, following experiment is carried out; the mortar specimens are put into the vacuum desiccator and evacuated. The loaded monomer in dropping funnel connected to the desiccator is slowly poured into the desiccator paying special attention to prevent air entering. When the mortar samples are completely submerged into monomer, slow stream of nitrogen gas is introduced to equilibrate the pressure in the desiccator with the atmosphere. In another method, after specimens are placed in the vacuum desiccator, plenty of the monomer is poured into the desiccator, the specimens are taken out at optional intervals, and weighed. The amount of monomer impregnated is calculated by the weight percent as to mortar.

b) Polymerization by radiation methods;

The mortar impregnated monomers are

weighed, wrapped with Al-foil and irradiated. The irradiated samples are unwrapped and put into a vacuum desiccator and fully evacuated to remove unpolymerized monomer or even oligomer. The increased weight is presumed as the weight of polymer loaded and the polymerization percentage is expressed as following

$$\text{Polymerization\%} = \frac{\text{Wt. of polymer loaded}}{\text{Wt. of monomer impregnated}} \times 100$$

c) Polymerization by thermal-catalytic methods;

The mortar samples impregnated with the monomers contained 2.2% benzoyl peroxide are wrapped with Al-foil and placed into the drybox which is kept at the optional temperature and time. The treatment after curing is as same as the above.

d) Polymerization by radiation-catalytic methods;

The mortar samples impregnated with various monomers contained 2.2% benzoyl peroxide as a catalyst and wrapped with Al-foil are irradiated.

3) Measurement of the Properties of Mortar-Plastic Composite.

a) Water absorptivity;

The M P C dried and weighed are immersed in water maintaining at 30°C. At the various intervals the specimens are reweighed. The increased weight of the specimen is expressed as percentage of the water absorptivity.

b) Acid resistance;

The dried specimens are immersed in 15% HCl solutions for 24 hours and 48 hours at the room temperature. The samples attacked by acid are again immersed in distilled water for 5 hours and washed with fresh water

twice. The samples are dried at the temperature of 100°C for 5 hours, and reweighed. The decrease in weight of the corroded samples below its original weight is expressed as percentage of the destruction with acid

c) Tensile strength;

In measuring the tensile strength of M P C, the splitting method is used¹⁾. The cylindrical testing specimen is placed so that it lies in the same axial plane between the top and bottom pressing plates of the compressive strength tester. Load is applied slowly on them. The splitting load is determined by reading the loading weight at the splitting point. The splitting tensile strength is calculated by the following equation⁹⁾

$$\delta_t = \frac{2p}{\pi dl}$$

where δ_t : Tensile strength (kg/cm²)

p : Splitting load (kg)

d : Diameter of sample (cm)

l : Length of sample (cm)

3. Results and Discussion

On the impregnation of styrene monomer, the maximum loadings show about 13% by

Table 1. The impregnation rate of styrene into mortar at 20°C.

Time (min.)	Impregnation, % (Wt.)	
	Atmospheric process	Vacuum process
5	6.5	10.0
10	6.9	11.6
20	6.9	12.5
30	6.9	—
60	7.0	11.3
180	7.0	12.4
1440	8.2	13.0

Specimen: $\phi 2.5 \times 5$ cm

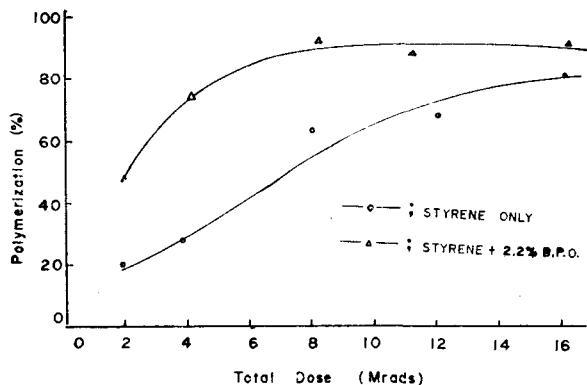


Fig. 1. Radiation-induced polymerization rate of styrene in mortar. (Dose rate: 8.4×10^4 rads/hr, Temp.: 20°C)

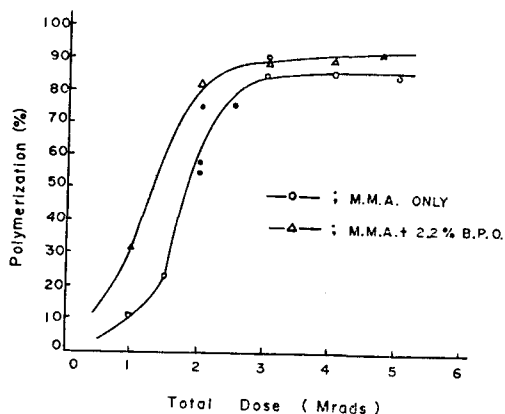


Fig. 2. Radiation-induced polymerization of M M A in mortar. (Dose rate: 1.0×10^5 rads/hr, Temp.: 20°C)

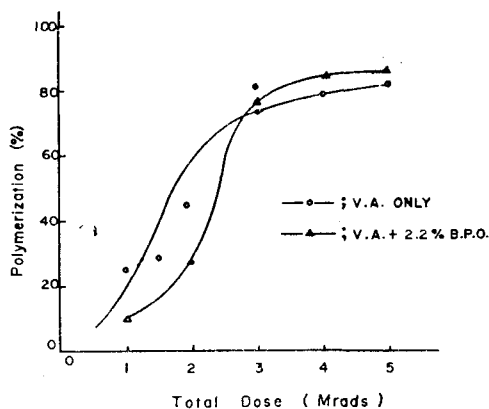


Fig. 3. Radiation-induced polymerization of vinyl acetate in mortar. (Dose rate: 1.0×10^5 rads/hr, Temp.: 20°C)

reduced pressure method and 8% by the simple dipping method respectively (Table 1). These correspond to double of the loadings in the case of concrete reported by Steinberg, *et al*⁶⁾. But it is easily comprehensible because a half of the concrete consist of coarse aggregate.

The soaking is almost equilibrated within ten minutes after the specimen were dipped into the monomer in both methods. However in the other experiment where larger specimens used, it was recognized that the reduced pressure method is also superior to the simple dipping method on the soaking velocity.

Almost same result would be expected for the other monomers because their physical properties (viscosity, specific gravity etc.) are almost as same as the styrene.

The radiation-induced polymerization rate of styrene, methyl methacrylate and vinyl acetate in mortar has been measured as a function of total dose under the dose rate of 8.4×10^4 , 1.0×10^5 and 1.0×10^5 rads/hr. respectively (Fig. 1-3). Under this condition about 15 Mrads for styrene and about 3 Mrads for M M A and V A are required to get more than 80% polymerization of the monomers. When benzoyl peroxide was a add to the monomers the polymerization rate were much increased except for the case of V A. The results almost agree with the case of concrete—M M A or concrete-styrene composite reported by Steinberg^{1) 6)}, though the rate of M M A is somewhat slower. The reason of the exceptional result of V A in adding the catalyst is not clear.

The thermal polymerization rate in mortar at 70°C for the monomers which included 2.2% benzoyl peroxide as a polymerization initiator are shown in Fig. 4. Generally, the rates under the condition were faster than those in

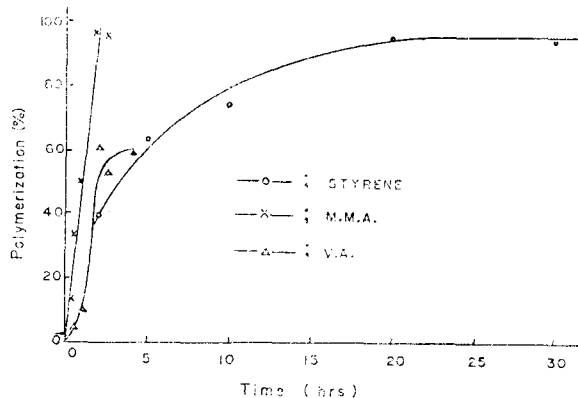


Fig. 4. Thermal polymerization of monomers in mortar at 70°C

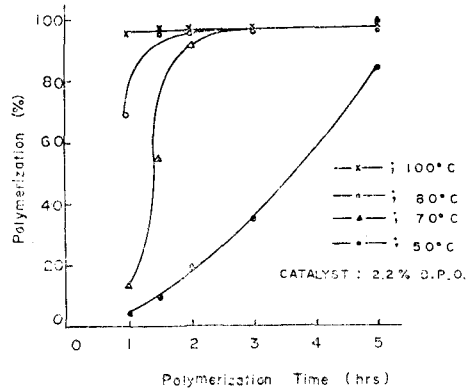


Fig. 5. Thermal polymerization rate of M M A in mortar at various temperatures.

the radiation curing and the order is M M A, V A Styrene. However, on the curing of V A, the reaction is ceased after about 60 percent conversion in spite of the fast rate in the first half period. From these points of view, M M A is the best monomer for thermal curing, but in treating and storing the M M A -benzoyl peroxide solution, a lot of caution must be paid to prevent the probable polymerization or explosion.

The effect of polymerization rate on the temperature in thermalcatalytic curing of M M A in mortar has been investigated in detail (Fig. 5). The higher temperature gets the faster polymerization. However, at high temperature the polymer products may be

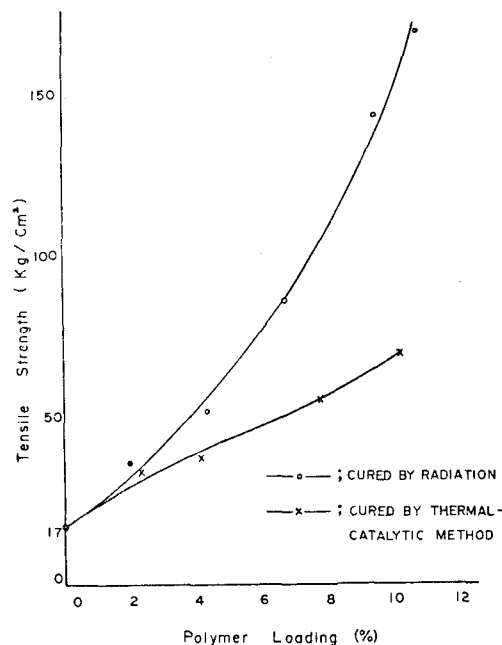


Fig. 6. Tensile strength v.s. polymer loading on mortar-polystyrene composite.

bubble or popcorned and shown poor mechanical properties.

The data on the reduction in water absorption by M P C are given in table 2. In general, the water absorptivity of M P C is much decreased in comparing with that of mortar, and is decreased by increasing polymer loading. The maximum reduction of more than 80% was obtained for the M P C containing about 10% of polymers. However, the method of polymerization and the kind of monomer do not give any significant differences.

These aspects and results also almost agreed with the case of concrete-styrene and concrete-M M A composite^{1, 7)} considering that the concrete has a half air void of the mortar in volume.

As shown in table 3, the acid resistance of M P C are also much improved comparing with that of control. The effect is more

Table 2. Water absorption of M P C

	Polymer loading, weight %	Radiation intensity, 10^4 rads/hr	Radiation dose, 10^6 rads	Water absorption, % (Wt.) at 30°C				
				2	5	24	48	(hr.) 336
Control	0	0	0	8.4	8.5	8.6	8.7	9.1
S R-A	7.1	8.4	20.1	1.3	1.5	2.0	2.3	3.6
S R-B	10.8	8.4	20.1	0.3	0.4	0.5	0.7	1.3
S T-A	7.4	0	0	0.6	0.9	1.2	1.6	2.8
S T-B	10.8	0	0	0.3	0.4	0.5	0.7	1.3
MR	5.0	4.0	4.8	1.5	1.7	2.0	2.2	2.8
MT	9.4	0	0	0.2	0.3	0.4	0.5	0.9
VR	7.9	4.0	7.7	2.7	2.8	2.9	3.0	3.4
VT	6.8	0	0	1.3	1.5	1.9	2.1	3.3

S: Styrene R: Radiation-catalytic M: Methyl methacrylate T: Thermal-catalytic V: Vinyl acetate

Table 3. Acid corrosion in 15% HCl at 20°C

Sample	Polymer loading, weight %	Radiation intensity, 10^4 rads/hr.	Radiation dose, 10^6 rads	Weight variations(%)	
				after 24 hrs.	after 48 hrs.
Control	0	0	0	-37.0	-65.0
S R	8.4	8.4	12.1	-6.0	-9.0
S T	8.0	0	0	-4.0	-6.0
M R	6.0	4.0	7.3	-30.0	-31.0
M T	8.5	0	0	-4.0	-5.5
V R	10.5	18.0	43.2	-17.6	-26.3
V T	9.8	0	0	-8.7	-10.5

S:Styrene R:Radiation-catalytic M:Methyl methacrylate T:Thermal-catalytic V:Vinyl acetate

advantageous than in the case of concrete because the acid corrosion of mortar is more severe than that of concrete.

On the view of polymerization method, the thermal curing method seems better than radiation curing for the resistance. For the M P C of styrene or M M A which loaded more than 8% polymer by the thermal curing, the erosion is reduced upto about 10 times of the control specimen. The cause may come from the accumulation of the polymers to the surface or near the outside of the M P C specimen by heating during polymerization. The acid resistance of V A-composite is somewhat lower than others.

The data of the tensile strength of the M P C are given in Fig. 6, 7 and 8, Compa-

red with $16\text{kg}/\text{cm}^2$ of the control specimen, the maximum increases over $160\text{kg}/\text{cm}^2$ can be obtained with styrene-and M M A-impregnated specimen.

In the case of C P C, the tensile strength was increased to about 4 times value of the control¹⁾. That is much less than those of M P C. This results may be due to the irregularity of the C P C structure. The concrete is consisted of a half of mortar and a half of pebble in which no polymer can be spreaded, so the tensile strength of C P C would be depended much on the adhesive strength of the polymer to pebble even the tensile strength of the pebble itself is very high. In other words, the poorer tensile strength of C P C comparing with that of M

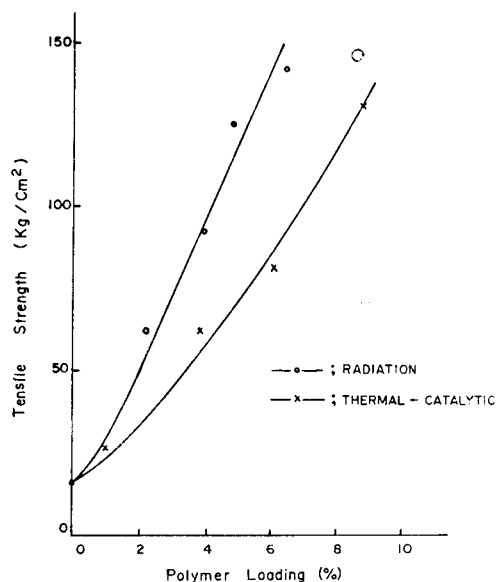


Fig. 7. Tensile strength v.s. polymer loading on M M A-M P C

P C would be mainly due to the poor adhesiveness of the polymers to the pebble.

It is also found that the M P C made by radiation curing process, in general, are much superior in tensile strength to those made by thermal process.

References

- 1) Steinberg, M. *et al*, "Concrete-polymer materials" first topical report. BNL 50134(T-509) and USBR GEN. REP. 40, (1968)
- 2) Kent, J.A., Winston, A.W. *et al*, USAEC

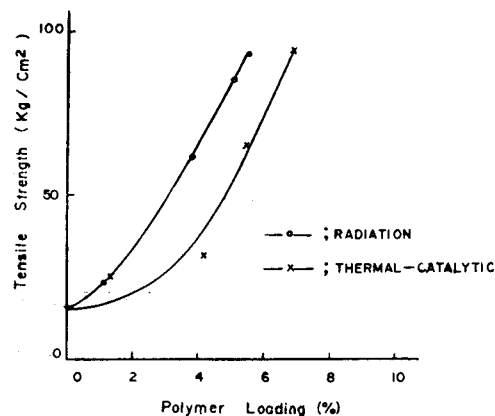


Fig. 8. Tensile strength v.s. polymer loading on vinyl acetate-M. P. C.

- Report, TID 7643, 335-344 (1968)
- 3) Kent, J.A., Winston, A.W. *et al*, USAEC Report, TID 18907, 9 (1963)
- 4) Siau, J.F., Meyer, J.A. *et al*, Forest. Prod. J. 15, 162) 1965)
- 5) Pyun, H.C., Kim, J.R. and Lee, K.H., J. Korean Nuc. Soc. 4. 23 (1972)
- 6) Steinberg, M. *et al*, "Concrete-polymer materials" second topical report, BNL 50218 (T-560) and REC OCE 70-1, (1969)
- 7) Dikeou, J.T., *et al*, "Concrete-polymer materials" third topical report, REC-ERC-71-6 and BNL 50275 (T-602), (1971)
- 8) Dikeou, J.T., *et al*, "Concrete-polymer materials" Fourth topical report, REC-ERC-2-10 and BNL 50328 (1972)
- 9) J.I.S. A 113.