

## Effects of TMPTMA and Silane on the Compressive Strength of Low-Temperature Cured Acrylic Polymer Concrete

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**ABSTRACT:** This study addresses the effects of additives on the compressive strength of low-temperature cured acrylic polymer concrete (PC). Three curing temperatures (0°C, -10°C, and -20°C) and five ages (6, 12, 24, 72, and 168 h) with two different types of additives [trimethylolpropane trimethacrylate (TMPTMA) and silane] were investigated. As a result, the compressive strength tended to decrease as the curing temperature decreased. The compressive strengths at 24 h were approximately 90% of those at 168 h at both curing temperatures of 0°C and -20°C, indicating that the rate of early age strength development was quite high even at a very low curing temperature range. The results of two-way variance analysis revealed that silane had a greater impact on the compressive strength than TMPTMA. About 13%–23% strength improvements with a 168-h compressive strength of over 80 MPa could be obtained at -20°C by adding silane. Furthermore, this study proposed optimum mixture proportions of acrylic PC that generate a working life of 50–70 minutes with a compressive strength of 80 MPa at subzero temperatures. The findings of this study are expected to be effectively used in field applications of acrylic PC, especially in the cold regions during winter season. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40939.

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### INTRODUCTION

Polymer concrete (PC), one of the representative concrete–polymer composites consisting of aggregates and polymeric binders, has been primarily used in cast-in-place applications such as repair of concrete pavements and airport runways as well as for precast products such as sewage pipes and manholes.<sup>1,2</sup> Recently, it is extensively used for transportation infrastructures subjected to severe environmental conditions especially bridge deck overlays, to enhance the durability and extend the life span.<sup>3</sup> PC has many advantages over ordinary cement concrete with respect to high strength, fast setting time, and excellent durability, and thus it has a sufficient competitive edge in terms of technical and economic aspects.

Binders used in PC include epoxy resin, unsaturated polyester resin, and acrylic resin, and exhibit great differences in physical and mechanical properties depending on their chemical components.<sup>4</sup> Among them, unsaturated polyester resin has an advantage in the unit cost compared with other types of resins, although its large volume changes due to setting shrinkage and thermal deformation limit its broad applications. On the other hand, PC using acrylic resin has a slightly higher unit cost but

provides superior strength characteristics, adhesion, volume stability, and weatherproofness and thus is widely used in repair of concrete pavements and manufacturing of precast products. Particularly, acrylic PC has better workability than other thermosetting resin PCs, because methyl methacrylate (MMA) monomer, the binder for acrylic PC, has quite low viscosity.<sup>5</sup>

Several former studies on acrylic PC are available. Fowler et al.<sup>6</sup> added trimethylolpropane trimethacrylate (TMPTMA) and tetraethylene diacrylate (TTEGDA) to MMA monomer and examined the strength developments at subzero temperatures while varying the contents of initiator [benzoyl peroxide (BPO)] and promoter [N,N-dimethylaniline (DMA)]. Their study found that, at subzero temperatures, the strengths tended to decrease even with the increased amounts of initiator and promoter. Haddad et al.<sup>7</sup> evaluated the strength characteristics of acrylic PC (with MMA as a binder and TMPTMA as a crosslinking agent) with various contents of initiator (BPO) and promoter (DMA) as well as curing temperatures (-1°C, 10°C, 21°C, and 38°C). They have reported that, as the curing temperature dropped from 38°C to -1°C, the flexural, bond, and compressive strengths were reduced by 8.1, 59.4, and 26.7%, respectively. Kobayashi et al.<sup>8</sup> stated that acrylic PC using polymethyl

**Table I.** Properties of MMA Monomer

Density (25°C)	Viscosity (20°C, mPa s)	Molecular weight (g/mol)	Appearance
0.9420	0.56	100	Clear colorless liquid

**Table II.** Properties of BPO

Melting point (°C)	Molecular weight (g/mol)	Appearance
104-105	242.23	White powder

methacrylate (PMMA) as a binder and TMPTMA as a crosslinking agent could achieve the 7-day compressive strength of over 80 MPa solely by modifying the contents of initiator (BPO) and promoter (DMA) at curing temperature of as low as 0°C to -20°C.

Most of the previous studies solely employed a crosslinking agent such as TMPTMA as an additive. Few research studies<sup>9</sup> have attempted to evaluate the effect of a coupling agent. Recognizing the current issue faced, the present study examines the effects of the both additives—a coupling agent and a crosslinking agent—on the compressive strength development of acrylic PC cured at subzero temperatures. The experimental variables were the contents of crosslinking agent (0, 2.5, and 5 phr) and coupling agent (0, 1, and 2 phr), and curing temperature (0°C, -10°C, and -20°C). Furthermore, this study attempts to provide the optimum mixture proportions of acrylic PC that meet the required compressive strength criterion at subzero temperatures, which is expected to benefit both cast-in-place and precast applications of acrylic PC especially in the cold regions.

## EXPERIMENTAL

### Polymeric Binder

**Acrylic Resin.** An acrylic resin was produced by dissolving PMMA into MMA monomer. PMMA is a powder highly soluble in MMA monomer which plays a role of thickening agent. MMA was made by oxidizing tert-butyl alcohol (TBA) in a gaseous state, thereby producing methacrylic acid (MAA), and then esterifying it with methanol. Table I shows the properties of MMA monomer used in the present study.

**Initiator.** BPO was used as an initiator. The interoxygen bonding of BPO is readily broken by heat and ultraviolet rays, producing radicals. It forms a new radical when combined with

**Table III.** Properties of DMT

Density (25°C)	Boiling point (°C)	Melting point (°C)	Molecular weight (g/mol)	Appearance
0.93	211	-15	135.21	Pale yellow liquid

**Table IV.** Properties of TMPTMA

Density (25°C)	Viscosity (25°C, mPa s)	Molecular weight (g/mol)	Appearance
1.061	44	338	Clear colorless liquid

**Table V.** Properties of Silane

Density (25°C)	Viscosity (25°C, mPa s)	Molecular weight (g/mol)	Appearance
1.045	5	248.35	Clear colorless liquid

monomers, which ultimately produces polymers. Table II presents the properties of BPO used.

**Promoter.** As a promoter, this study employed DMT. This promotes polymerization even at low temperatures. The detailed properties of DMT used are presented in Table III.

### Additives

**Crosslinking Agent.** A crosslinking agent is used to create a network-structured high molecule polymer by bridging the voids among molecules in a linear-structured polymer. TMPTMA, which is a tri-functional monomer that increases the crosslink density, is an additive ensuring rapid hardening reactions through free radical polymerization. Table IV displays the properties of TMPTMA used in this study.

**Coupling Agent.** Because PC is formed by combining polymer matrices (i.e., organic compound) with aggregates (i.e., inorganic mineral substance), it typically has weak chemical adhesiveness. Silane, a coupling agent, improves the dispersibility of PC since one side of its molecule is combined with mineral substances while the other side is combined with polymer matrices. As a result, the interfacial adhesion with an inorganic material is greatly improved, enhancing overall mechanical properties of PC. Table V summarizes the properties of silane employed in this study.

### Aggregate and Filler

The quality of aggregates used in PC is comparable to that used in conventional portland cement concrete. Since the adhesion between polymeric binders and aggregates becomes weaker as the aggregates absorb moisture, it is essential to keep the moisture content less than 0.1%. Table VI shows the properties of aggregates used.

**Table VI.** Physical Properties of Aggregate

Grain size (mm)	Apparent specific gravity	Unit weight (kg/m <sup>3</sup> )	Fineness modulus	Moisture content (%)	Organic impurities
0.08-8	2.64	1648	3.09	<0.1	Nil

**Table VII.** Physical Properties of Heavy Calcium Carbonate Filler

Specific gravity (g/mL)	Absorption (mL/g)	Moisture content (%)	pH	Mean grain size ( $\mu\text{m}$ )	Retained percentage of 325 mesh sieve
0.75	0.20	$\leq 0.3$	8.8	13	0.03

Also, in PC, filler is used to bulk up the polymeric binders and fill the voids among aggregate particles.<sup>10</sup> The present study employed a single source of heavy calcium carbonate filler as it is inexpensive and provides good workability. The filler had a grain size of 1–30  $\mu\text{m}$ , fineness of 2500–3000  $\text{cm}^2/\text{g}$ , and moisture content of less than 0.3%. Tables VII and VIII present the physical properties and chemical compositions of heavy calcium carbonate filler used.

### Mixture Proportions

The optimum content of polymeric binder was selected based on flow tests. In the testing program, a MMA to PMMA ratio and binder to filler ratio were fixed to 80 : 20 and 1 : 1.5 by weight, respectively. Based on the results of preliminary flow test, the optimum content of polymeric binder was found to be 11–12 wt % with the corresponding flow values of 110–123 mm; when the flow value fell between 100 and 105 mm, severe segregation was observed, while excessive bleeding occurred when the flow value was greater than 130 mm. Accordingly, all the mixtures tested in this study had 11 wt % of polymeric binder. Table IX shows the binder formulations and mixture proportions of acrylic PC evaluated in this study. Here, the DMT content varied depending on the TMPTMA content in order to keep the working life to approximately 60 minutes.

### Compression Test

Three cylindrical specimens ( $\text{Ø}5 \times 10 \text{ cm}$ ) were prepared for each experimental variable and then were cured at various temperatures ( $0^\circ\text{C}$ ,  $-10^\circ\text{C}$ , and  $-20^\circ\text{C}$ ). Subsequently, compression testing was conducted in accordance with 1995 TC-CPT of the RILEM<sup>11</sup> (*Method of Test for Compressive Strength of Polymer Concrete and Mortar PC-5*) using a 20-ton UTM (Instron 8502) at 6, 12, 24, 72, and 168 h. The testing was performed at standard temperature of  $23^\circ\text{C} \pm 2^\circ\text{C}$ .

## RESULTS AND DISCUSSION

### Effect of Curing Temperature

Figure 1(a–c) presents the results of compression test for different silane contents. For sample identification, the first number in the legend box denotes the curing temperature ( $^\circ\text{C}$ ) and the number next to the curing temperature represents the TMPTMA content (phr). The trend of the results was nearly

**Table VIII.** Chemical Compositions of Heavy Calcium Carbonate Filler

CaO (%)	$\text{Al}_2\text{O}_3$ (%)	$\text{Fe}_2\text{O}_3$ (%)	$\text{SiO}_2$ (%)	MgO (%)	Ignition loss (%)
53.7	0.25	0.09	2.23	0.66	42.4

consistent in that most of the compressive strength developed within the first 24 h after mixing. Thereafter, the rate of compressive strength development was prominently reduced, showing a quite small strength increment.

Also, the results clearly indicate that the compressive strength development was affected by curing temperature. The ratio of compressive strength at 24 h to that at 168 h was 93.5% on average at  $0^\circ\text{C}$  but was decreased to 86.7% at  $-20^\circ\text{C}$ . Also, about 4–6 MPa of compressive strength reduction was observed as the curing temperature decreased from  $0^\circ\text{C}$  to  $-20^\circ\text{C}$ . These findings demonstrate that the compressive strength of acrylic PC decreases with the reduced curing temperature, as conventional cement concrete does. In addition, the obtained result was similar to that of the previous studies by Haddad et al.<sup>7</sup> and Kobayashi et al.,<sup>8</sup> which reported that the compressive strength of acrylic PC is substantially dependent on curing temperature.

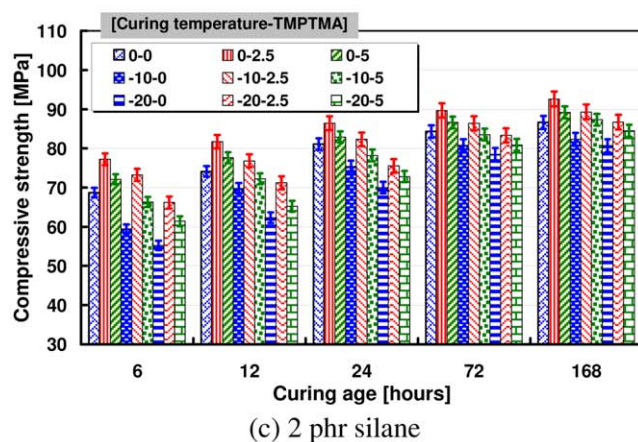
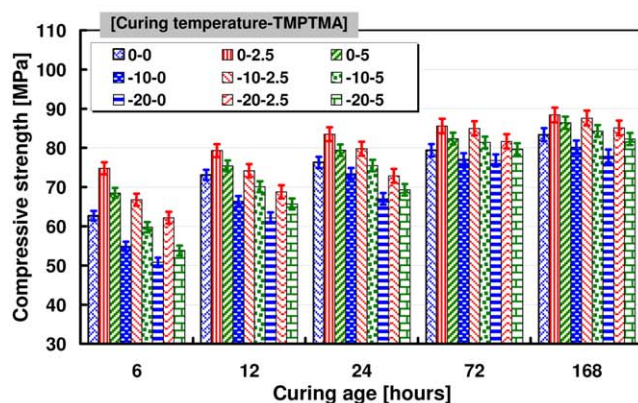
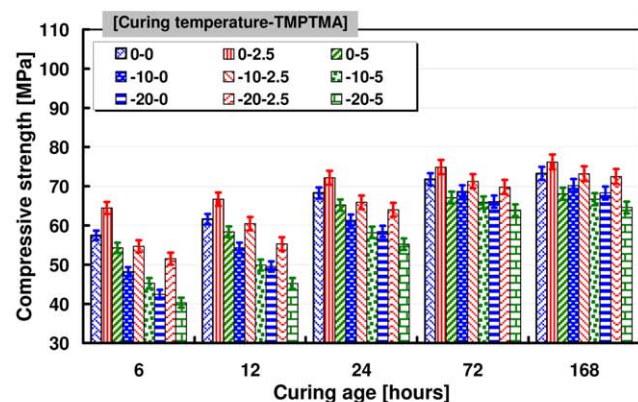
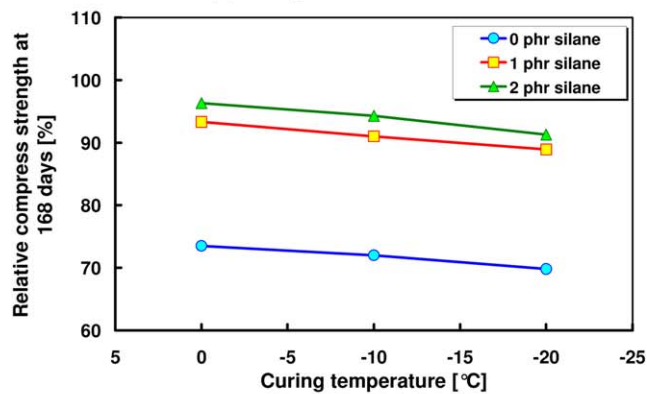
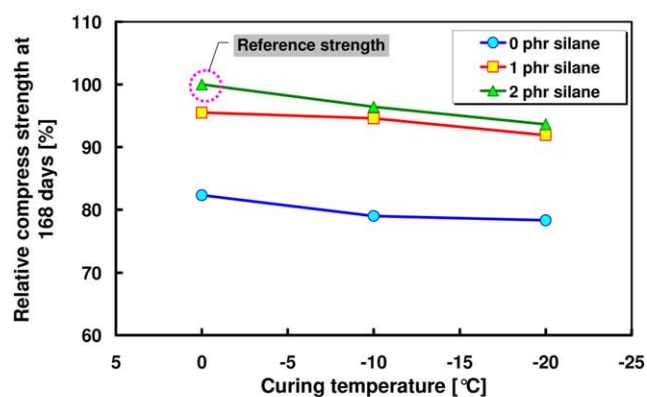
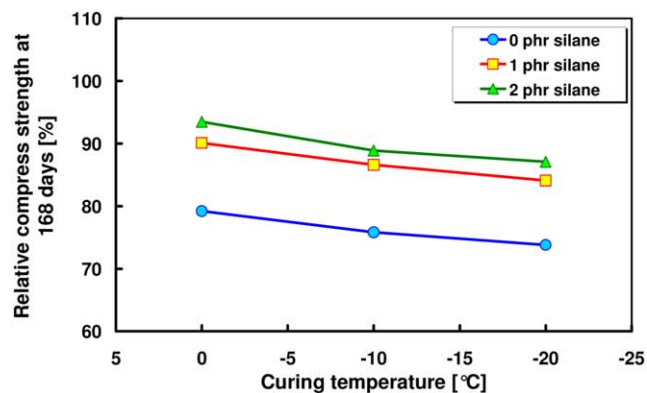
Figure 2 illustrates the variations of 168-h relative compressive strength with curing temperature. In this work, the relative compressive strength was defined as the compressive strength ratio to the highest compressive strength (achieved when the curing temperature was  $0^\circ\text{C}$ , TMPTMA content was 2.5%, and silane content was 2 phr). As the results show, the relative compressive strength tended to monotonically decrease as the curing temperature decreased. The differences between the highest and lowest values ranged from 4% to 7% for the fixed silane content. It should be noted that there were large differences between the cases with and without silane, which implies that silane had a significant effect on the compressive strength of acrylic PC.

### Effect of TMPTMA

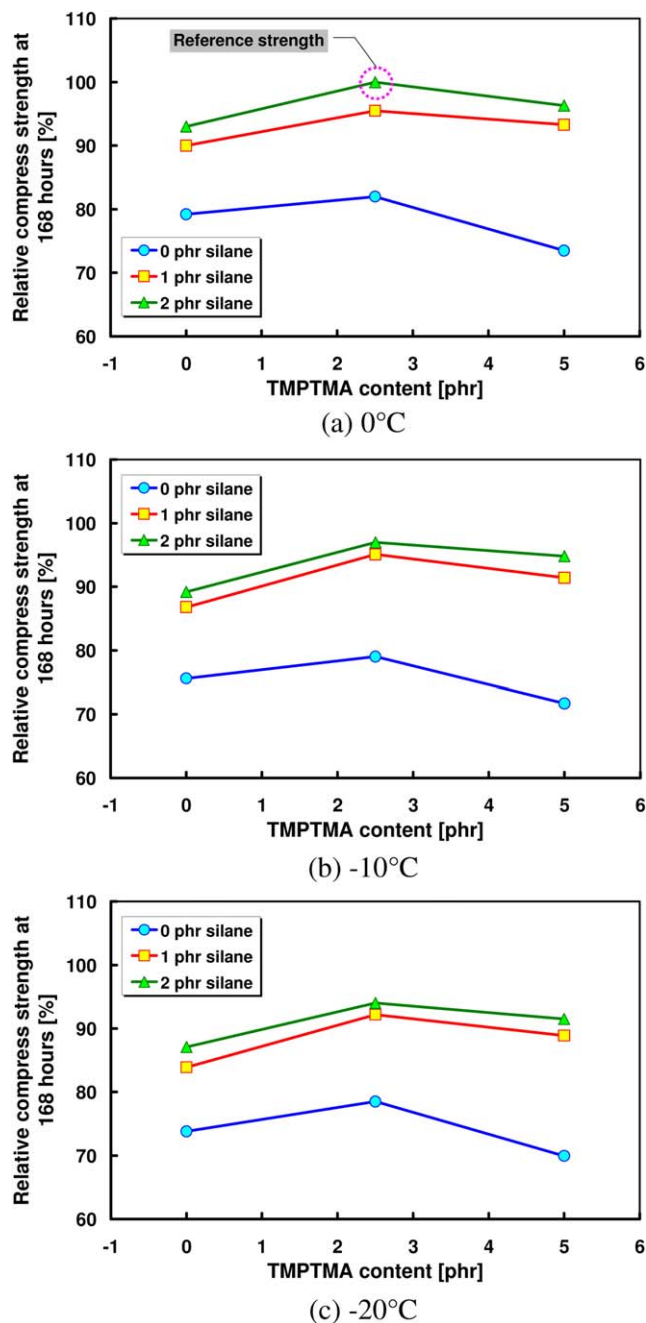
Recall Figure 1 that shows the influence of TMPTMA on the compressive strength of acrylic PC. Note that the trend was not as consistent as the effect of curing temperature. As the TMPTMA content increased from 0 to 2.5 phr, there was an increasing tendency in the relative compressive strength. However, as the TMPTMA increased beyond 2.5 phr, the compressive strength tended to decrease in all cases. Especially, when silane was not added, the compressive strength with 5 phr TMPTMA was even lower than that without TMPTMA. This behavior indicates that the excessive use of TMPTMA rather caused a negative effect on the compressive strength. Two possible mechanisms can be considered causes of this behavior. First, the excessive use of TMPTMA might increase the crosslink density, making the matrices more brittle. Moreover, some imperfect synthesis might occur due to the strong interfacial repulsion among the phases, which resulted in phase separation. Based on the shape of specimen failure observed, it appears that phase separation is a more probable cause.

**Table IX.** Binder Formulations and Mixture Proportions of Acrylic PC

Binder content (wt %)	Binder formulation (wt %)						Aggregate (wt %)
	MMA : PMMA	TMPTMA (phr <sup>a</sup> )	Silane (phr)	BPO (phr)	DMT (phr)	Filler (wt %)	
11	80:20	0	0		1.5	16.5	72.5
		2.5	1	2	1.0		
		5.0	2		0.5		

<sup>a</sup>Parts per hundred parts of resin.**Figure 1.** Results of compression test. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]**Figure 2.** Curing temperature versus relative compressive strength at 168 h. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]





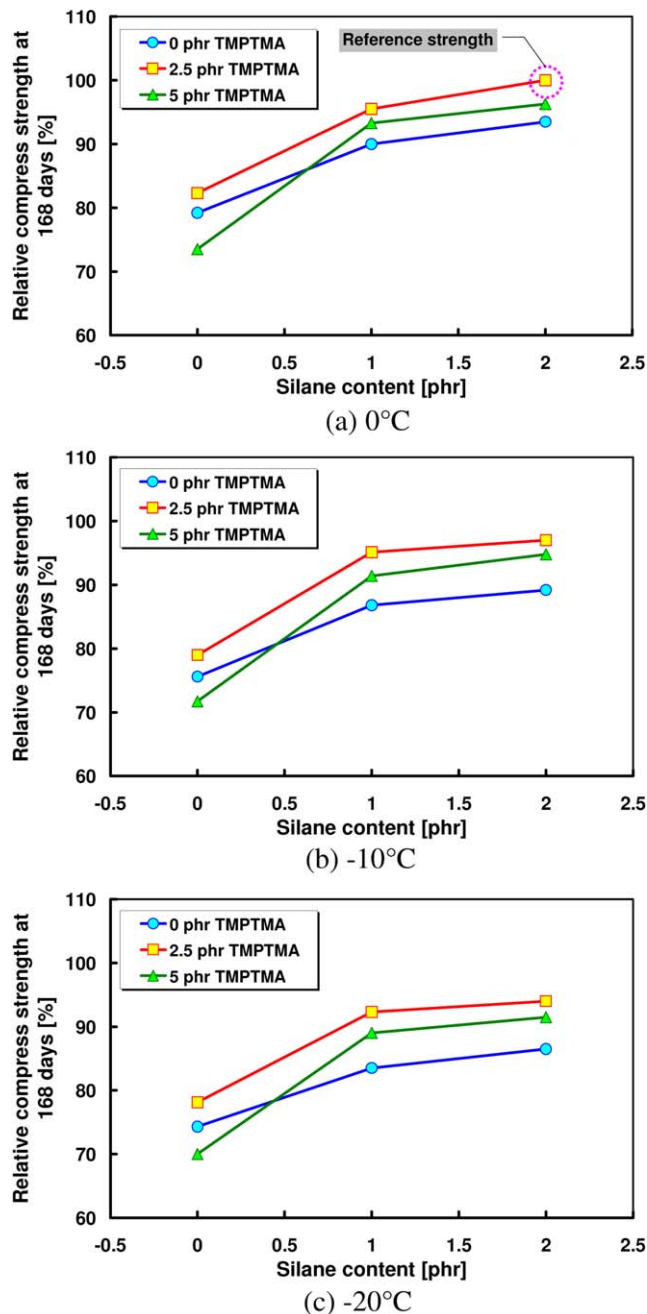
**Figure 3.** TMPTMA content versus relative compressive strength at 168 h. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

Figure 3 presents the changes in 168-h relative compressive strength for various TMPTMA contents. The results revealed that, as aforementioned, the relative compressive strength with 5 phr TMPTMA was smaller than that with 2.5 phr. Also, it was found that the difference in relative strength between 2.5 and 5 phr became greater as the TMPTMA content increased. The obtained result was somewhat different from the previous study by Kobayashi et al.,<sup>8</sup> which stated that compressive strength of acrylic PC was highest for 5 phr TMPTMA at  $-20^{\circ}\text{C}$ . The reason for this difference is most likely because the former study

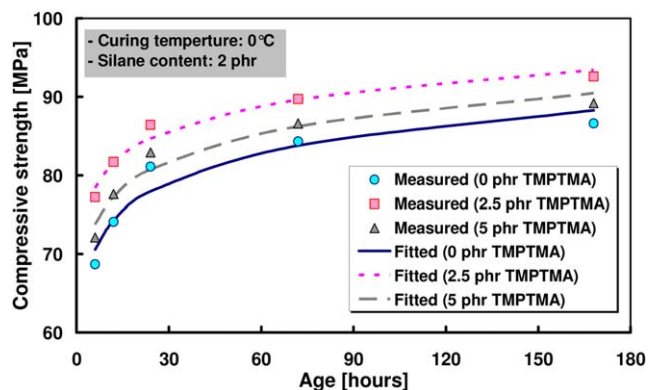
incorporated only 5% PMMA in MMA monomer, while the current study allowed up to 20% PMMA.

#### Effect of Silane

The compressive strength of acrylic PC was closely related to the silane content as previously shown in Figure 1(a–c). Overall, the compressive strength tended to noticeably increase upon addition of silane. From the results, it can be also found that the 168-h compressive strength of over 80 MPa could be obtained even at subzero temperature as low as  $-20^{\circ}\text{C}$  solely by adding silane.



**Figure 4.** Silane content versus relative compressive strength at 168 h. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 5.** Example fitting curves for estimating compressive strength development. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

Figure 4 depicts the effect of silane content on the 168-h relative compressive strength. Note that silane was quite effective in improving the compressive strength by up to 13%–23%.

However, there was no considerable discrepancy in relative compressive strength between the 1 and 2 phr cases. This phenomenon demonstrates that no more than 2 phr silane is essentially required for strength improvements of acrylic PC. Moreover, it can be seen from the results that the relative compressive strength was highest for 2.5 phr TMPTMA, and when the silane content was 1–2 phr, the relative compressive strength with 5 phr TMPTMA became greater than that without TMPTMA. This finding implies that the effect of silane became more pronounced with addition of TMPTMA. The mechanism behind this behavior needs to be further investigated. The outcome of this study is comparable to that of the previous studies by Yeon et al.,<sup>12</sup> which found that the addition of 1 phr silane increased the compressive strength of polyester PC by 5.1%, and by Chmielewska et al.,<sup>13</sup> which showed that vinyl ester polymer mortar yielded about 15% compressive strength increase with addition of 1 phr silane. Also, it was even similar to the result of the former study by Ohama et al.,<sup>5</sup> which reported that PMMA PC experienced approximately 10%–20% compressive strength increase upon addition of silane.

**Table X.** Parameters and Correlation Coefficients for Fitting Curves

Curing temperature (°C)	TMPTMA content (phr)	Silane content (phr)	Parameters		$R^2$
			$a$	$b$	
0	0	0	4.8447	50.168	0.926
		1	5.8631	55.557	0.907
		2	5.3160	61.040	0.924
	2.5	0	3.6745	58.513	0.932
		1	3.8772	69.250	0.944
		2	4.5150	70.300	0.965
	5.0	0	4.1941	48.281	0.909
		1	4.9582	61.706	0.942
		2	5.0138	64.778	0.950
-10	0	0	6.8235	37.537	0.954
		1	7.2027	46.119	0.901
		2	6.6708	51.152	0.901
	2.5	0	5.5704	46.342	0.956
		1	6.0857	58.165	0.947
		2	4.8864	65.127	0.975
	5.0	0	6.8799	34.007	0.943
		1	6.9837	50.677	0.926
		2	6.2226	56.523	0.975
-20	0	0	7.9705	30.091	0.949
		1	8.0199	39.904	0.926
		2	7.8578	42.851	0.961
	2.5	0	6.6028	40.361	0.954
		1	6.9195	50.814	0.986
		2	6.2687	55.448	0.992
	5.0	0	7.8714	27.305	0.936
		1	8.3101	42.154	0.943
		2	7.2936	48.373	0.983

**Table XI.** Results of Two-Way ANOVA for 0°C Curing Temperature

Variable factor	Sum of squares	Degree of freedom	Root mean square	F-ratio	P-value	F-critical value
TMPTMA	41.7355	2	20.8677	3.6731	0.1242	6.9442
Silane	481.4489	2	240.7244	42.3720	0.0020	6.9442
Residual	22.7244	4	5.6811	-	-	-
Total	545.9089	8	-	-	-	-

**Table XII.** Results of Two-Way ANOVA for -10°C Curing Temperature

Variable factor	Sum of squares	Degree of freedom	Root mean square	F-ratio	P-value	F-critical value
TMPTMA	52.5955	2	26.2977	4.7688	0.0873	6.9442
Silane	465.7422	2	232.8711	42.2293	0.0020	6.9442
Residual	22.0577	4	5.5144	-	-	-
Total	540.3956	8	-	-	-	-

**Table XIII.** Results of Two-Way ANOVA for -20°C Curing Temperature

Variable factor	Sum of squares	Degree of freedom	Root mean square	F-ratio	P-value	F-critical value
TMPTMA	54.3800	2	27.19	5.0258	0.0810	6.9442
Silane	421.9800	2	210.99	39.0000	0.0023	6.9442
Residual	21.6400	4	5.41	-	-	-
Total	498.0000	8	-	-	-	-

### Regression Analysis of Compressive Strength Development

The measured compressive strength evolution can be fitted in a logarithmic function considering the content of additives and curing temperature as shown in Figure 5 with an example. The basic form of a regression equation can be written as  $f'_c = a \ln(t) + b$ ; where  $f'_c$  is the compressive strength [MPa],  $a$  and  $b$  are the fitting parameters for regression curve [-], and  $t$  is age [h]. The fitting parameters for regression curves and the corresponding correlation coefficients evaluated are summarized in Table X. As the table shows, the  $R^2$  for all analysis cases were at least 0.90, indicating that the proposed regression model was statistically quite confident. The information provided in this study is expected to be effectively used for estimating the compressive strength development of acrylic PC for various curing temperatures and additive contents.

### Statistic Analysis of Experimental Data

In order to investigate the effects of TMPTMA and silane on the compressive strength of acrylic PC at 168 h, a two-way analysis of variance was performed in terms of each of the three curing temperatures. Tables XI–XIII present the results of two-way variance analysis. The data shows that TMPTMA had no statistically significant correlation to the compressive strength as the  $F$ -ratios were lower than the  $F$ -critical values, and also the  $P$ -values were higher than 0.05, that is, the significance level. This result was obtained because the compressive strength began to decrease as the content of TMPTMA became higher than 2.5 phr. On the other hand, the analysis results for silane showed that the  $F$ -ratios were much higher than the  $F$ -critical values and the  $P$ -values were much lower than 0.05, which implies that silane had a high statistical significance.

**Table XIV.** Optimum Mixture Proportions for Acrylic PC at Subzero Temperatures

Curing temperature (°C)	Binder content (wt %)	Formulation of binder (wt %)					Filler (wt %)	Aggregate (wt %)
		MMA:PMMA	TMPTMA (phr)	Silane (phr)	BPO (phr)	DMT (phr)		
0			0	1		1		
-10	11	80:20	2.5	1	2	0.5	16.5	72.5
-20			2.5	1		1		
			5			0.5		

### Optimum Mixture Proportions

The optimum mixture proportions of acrylic PC were found based on the experimental results obtained in this study. As previously described, curing temperatures were set to 0°C, -10°C, and -20°C considering the environmental conditions in the cold regions during winter season. TMPTMA and silane were chosen as additives. An optimum working life was selected as 50–70 minutes recognizing the typical working environment of field practices. A target 168-h compressive strength was 80 MPa. Table XIV exhibits the optimum mixture proportions of acrylic PC that meet all the selected criteria empirically determined through the current study.

### CONCLUSIONS

On the basis of the results of laboratory testing and statistic analysis conducted in this study, the following concluding remarks can be drawn:

- a. Most of the compressive strength of acrylic PC evolved within 24 h after mixing, even at subzero temperatures. This finding demonstrates that the rate of strength development is significantly high at very early ages.
- b. As expected, the compressive strength of acrylic PC tended to decrease as the curing temperature decreased.
- c. Silane had a remarkable effect on the compressive strength of acrylic PC. By adding silane, a compressive strength of 80 MPa or higher could be achieved even at subzero temperature as low as -20°C.
- d. The compressive strength tended to decrease as the content of TMPTMA increased beyond 2.5 phr. This is because the excessive use of TMPTMA yielded intense interfacial repulsive forces among the phases, which led to phase separation.
- e. A logarithmic function reliably fitted the compressive strength development of acrylic PC considering the content of additives and curing temperature.
- f. The results of two-way analysis of variance showed that silane had a remarkable effect on the compressive strength development of acrylic PC, whereas TMPTMA did not.
- g. The optimum mixture proportions that meet a working life of 50–70 minutes as well as a compressive strength of 80 MPa at subzero temperatures were proposed for the use of acrylic PC in field applications especially in the cold regions during winter season.

The current study was limited to the macroscopic interpretations of experimental results. For more in-depth evaluation of the additive effects on mechanical properties of acrylic PC, further research on microstructural characterization needs to be conducted.

### ACKNOWLEDGMENTS

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