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ANALYSIS AND EXPERIMENTAL TESTING OF PHYSICAL AND MECHANICAL PROPERTIES OF FLOORING EPOXY

Abstract

Epoxy resins are a class of reactive oligomers and polymers with epoxide groups. Depending on the type of epoxy and the hardener used, the curing (hardening) process can be regulated. Consequently, epoxy resins can be characterized by the "working time" or pot-life - the time it provides for certain technological operations to be performed with it. In the hardened state, epoxies are characterized by high strengths and high chemical resistance. Among other applications, they are widely used as materials for industrial floors, and for waterproofing. Laboratory optimization tests have shown that high strengths can be achieved with a lower density, and that the use of quartz filler does not necessarily improve the strength of the epoxy.

Keywords: epoxy, laboratory tests, physical and mechanical properties

АНАЛИЗА И ЕКСПЕРИМЕНТАЛНО ИСПИТИВАЊЕ ФИЗИЧКИХ И МЕХАНИЧКИХ СВОЈСТАВА ЕПОКСИДА ЗА ПОДОВЕ

Сажетак

Епоксидне смоле су врста реактивних олигомера и полимера који поседују епоксидне групе. У зависности од типа епоксида и употребљеног очвршћивача, процес неге (очвршћавања) може се регулисати. Последично, епоксидне смоле се могу карактерисати "радним временом" (pot-life), односно временом које је потребно да се обаве одређене технолошке операције. У очврслом стању, епоксиди се карактеришу високом чврстоћом и високом хемијском отпорношћу. Између осталих намена, они се широко употребљавају за индустријске подове, као и за остваривање водонепропустљивости. Оптимизацијом и лабораторијским испитивањима показано је да се са нижом запреминском масом могу постићи сличне чврстоће, као и да употреба кварцног пуниоца не резултира обавезно побољшањем чврстоће.

Кључне ријечи: епоксиди, лабораторијска испитивања, физичка и механичка својства

1. INTRODUCTION

The amounts of epoxy resins manufactured and consumed are insignificant in comparison with polyethylene, polypropylene and polystyrene. Even when resins such as polyesters and polyurethanes are considered, the amounts of epoxy resins produced are smaller. But in terms of complexity of technology, variety and breadth of application, epoxy resins are surely superior to all other plastics and resins. There is hardly an industry in which these resins are not used, and are especially used in construction [1]. They find applications in electrical and electronic devices and oil wells, in space satellites and stained-glass windows, on roads and bridges and in computers, in skis, and in supersonic aircraft. They form the basis of high-performance paints which are used on ships, automobiles and as food can linings. They are used in the factory, the artist's studio, the laboratory and in the home.

Epoxy resins can be used as adhesives, sealing compounds, casting resins, dipping compounds, molding powders, paints and varnishes. powder coatings, flooring and anti-skid surfacing, and as the resin matrix in reinforced composites. When in these various forms, they can be manipulated by hot or cold spraying, brushing, roller coating and all the other paint application methods, knifing, dipping, pouring, high- and low-pressure compression molding and injection molding. The reasons for this diversity of applications lie in the fundamental properties of the resins. Thus, during cure, no volatiles or other by-products are formed and volumetric shrinkage is very small of the order of -2%. The fully cured epoxy resin systems have the well-known properties of outstanding adhesion to many substrates, chemical resistance, toughness, mechanical strength, and high electrical resistance. Many variations of these properties can be achieved by adjustments in the formulation used.

1.1. BASIC EPOXY RESIN TECHNOLOGY

Epoxy resins, also known as epoxide or ethoxyline resins, contain the epoxy group which is the base of their structure. When manufactured, the resins are either liquids or solids and contain, on average, two epoxy groups per molecule. The resins are thermoplastic in this physical state, that is, they can be repeatedly softened by heat and hardened by cooling.

However, the essential feature of epoxy resin technology is the conversion of the resin into a hard, infusible three-dimensional network in which the resin molecules are crosslinked together by means of strong covalent bonds. This conversion can be termed "polymerization", but is more commonly called "curing" or "hardening" of the resin. The reagents that influence this change are known as "curing agents" or "hardeners". It is in this hardened (cured) form that the resins are almost always used; in the uncured, "non-crosslinked" state they are of limited use.

Hardening is an irreversible change and once the resin has been hardened, it cannot be recovered again in its original form. Hardening can be slowed down, stopped or speeded up, but it cannot be reversed. Hence resins fall into the category of thermosetting polymers, which, once polymerized, cannot be re-used by melting and reprocessing. Continued heating of a thermoset merely leads to softening and eventually degradation and breakdown of the material.

Some curing agents will react with the resins at room temperature or below, while others require heat to affect the polymerization, or the presence of the solvent. The curing reaction is exothermic, that is, heat is evolved during the crosslinking process, which causes an increase in the temperature of the system, sometimes 150-200°C or above.

The temperature level reached in any particular example will depend not only upon the reactivity of the resin and curing agent, but also upon the temperature of the reactants and of their surroundings, that is, upon the rate at which polymerization is occurring and the rate at which the heat evolved is being dissipated to the surroundings. Clearly, the ratio of the surface area to the mass of the reactants is important, as a large surface area would allow more heat to be dissipated compared with a smaller surface area for the same mass. This situation occurs if a mixture is being used as an adhesive rather than for casting purposes.

In practice, the attention is paid to ensure that the increase in temperature of a bulk mixture of resin and curing agent is not excessive. In the absence of such control, bubbling, cracking, charring and even complete degradation of the resin could occur in severe cases.

The time taken from the initial mixing of the resin and curing agent to the point when the viscosity of the mixture has become so high as to render the mix unusable is called the "pot life' of the system. This time is therefore the practical working life of the mix, during which the material must be applied efficiently. To a large extent, the pot life is influenced by the same factors that affect the exotherm, and pot lives can vary from a few seconds to several weeks, such is the wide range of possibilities with eposy resin formulations.

To a large extent, the pot life is influenced by the same factors that affect the exotherm reaction, and pot lives can vary from a few seconds to several weeks, such is the wide range of possibilities with epoxy resin formulations. The pot life is also dependent upon the intended application of the system; thus, a viscous mixture may still be pourable into a mold, but would not be suitable for glass-fiber impregnation in a laminating process.

The simple resin-curing agent combination alone seldom provides a material with all the properties required for use in a given application, and other materials must be added so as to modify the properties of the cured resin or to make it cheaper. The correct choice of the types and amounts of the different components of an epoxy formulation is fairly precise, difficult and important task because the final properties and eventual performance of the system depend upon it. The various classes of materials that can be added to the resin and curing agent combination are in Table 1.

Table 1.Some of the different classes of materials that can be added to the resin-hardener mixture

Diluents	Liquids used to reduce the viscosity of the mixture
Inert filers	Mainly used to make the system cheaper and to modify the physical and mechanical properties such as thermal conductivity and expansion, hardness and compressive strength
Fire retardants	Techniques employed to improve the fire-retardant properties of the cured resins mostly involve the incorporation of bromine or chlorine atoms into the system, usually by utilizing halogenated epoxy resins or chlorinated curing agents
Resinous modifiers	These systems have enhanced properties in certain respects over the properties of the individual, separate resins
Cure accelerators	Certain simple substances can increase the rate of reaction between the epoxy resins and some curing agents

Some modifying materials frequently perform more than one function at the same time [2].

1.2. ADVANTAGES OF USING EPOXY RESINS

For the designer and architect, the problem of material selection is often very complex and difficult. There is a bewildering variety of materials and processes available, and it is not easy to obtain a balanced and unbiased assessment of the merits and demerits of any individual material or group of materials from the information available. Load-bearing materials can illustrate the breadth of choice which confronts the design engineer. The range of suitable materials can extend from the traditional structural materials such as stone, wood, metals and concrete to the thermoplastic and thermosetting polymers, and to composite materials in which the stiffness and/or strength of a material is greatly enhanced by its combination with another material.

The relative advantages of epoxy resins have been discussed by a number of authors elsewhere, for example by Alexander [2], [3] and Miller [4]. Besides the basic advantages of epoxy resins, a question has to be answered why epoxy resins have been used for certain applications instead of another polymer or traditional material; what advantages epoxy resins have over other materials, and how these advantages can best be exploited. Plastics in general offer a combination of properties that are often superior to other materials in many respects. For example, plastics can have:

- The ability to be molded directly into complex shapes,
- High electrical resistance,
- High corrosion and chemical resistance,
- Optical clarity,
- High strength to weight ratios,
- Attractive surface texture and color.

In a similar way to metals, certain plastics can also be processed by mass production methods in which the product is made with consistent and reproducible accuracy. In contrast, large structures in glass-reinforced plastics are laid up by hand as single units.

1.3. COMPARATIVE ANALYSIS OF PROPERTIES

Some general physical and mechanical properties of an unfilled casting epoxy resin (such as specific gravity, thermal conductivity, coefficient of thermal expansion, hardness, tensile and compressive strength, elongation at break, modulus of elasticity) are given in Table 2.

Table 2.General physical and mechanical properties of an unfilled casting epoxy resin [3,4]

Property	***Value***
Specific gravity	*1.2-1.3 g/cm^3*
Thermal conductivity	*4-5 x 10^{-4} cal/cm/s/deg C*
Coefficient of thermal expansion	*5-9 x 10^{-5} cm deg C/cm*
Hardness	*100-110 (Rockwell M scale)*
Ultimate tensile strength	*28-91 MPa*
Elongation at break	*1-8 %*
Modulus of elasticity	*1.4-3.5 GN/m^2*
Ultimate compressive strength	*70-210 MPa*

A unique approach to the comparison of structural materials was made by Alexander [3,4] who compared the cost of "l ton of strength" for various structural materials. Assuming an average manufactured cost of an epoxy-woven glass laminate of £4000/ton, the cost per ton per unit tensile strength was calculated to be 2.44 £/cm^2. On the same basis, the cost of structural steel was 0.19 £/cm^2, the cost of concrete 0.19-0.24 £/cm^2, timber 0.32-0.45 £/cm^2, copper castings 0.38-0.45 £/cm^2 and brass strip 1.1 -1.92 £/cm^2. Clearly, on the basis of this analysis, there was no clear reason why an epoxy laminate, or any other polymeric system, should replace steel or reinforced concrete at that time (1967). The data used to arrive at these costs should naturally be brought up to date if this approach is to be used today by an engineer, but on the contrary, this approach raises a question why epoxy or polyester-glass laminated pipes, for example, have ever replaced steel pipes. Clearly, it is not sufficient for material selection to be based on each property alone. A cost-benefit analysis should be carried out on each property of the material and additionally, on the other relevant aspects of the manufacture and use of the particular item under consideration.

The versatility of the resins and the advantages that can result from using them are really unique. The leading features of epoxy resins are:

- The ability to be cured rapidly or slowly over a wide range of temperatures,
- The ability to be processed by a large number of different techniques,
- The absence of volatile by-products formed during the curing reaction,
- Low shrinkage during cure,
- Excellent adhesion to many different substrates,
- A high level of mechanical strength, which is retained at elevated temperatures,
- Outstanding toughness,
- Good electrical properties,
- Excellent chemical resistance.

2. EXPERIMENTAL TESTING OF PHYSICAL AND MECHANICAL PROPERTIES OF EPOXY

The experimental tests were conducted on the epoxy resin "MC-DUR 1320 VK", of German production. As a transparent epoxy resin, it is recommended by the manufacturer for use in parking lots and industrial facilities. Technical values and product characteristics for MC-DUR 1320 VK are given in Table 3.

Table 3.Technical data for the applied epoxy resin [7]

Property	***Unit***	***Value***	***Comments***
Mixing ratio	*mass fractions*	*5:1*	*base : hardener content*
Density	*g/cm^3*	*approx. 1,5*	-
Viscosity	*mPa's*	*approx. 2400*	*at +20 ^{0}C and 50% rel. humidity*
Working time	*minutes*	*approx. 45*	*at +20 ^{0}C and 50% rel. humidity*
Accessible after	*hours*	*approx. 12*	*at +20 ^{0}C and 50% rel. humidity*
Resilient after (full)	*days*	*7*	*at +20 ^{0}C and 50% rel. humidity*
Application conditions	*^{0}C*	*>10- <30*	*air and substrate temperatures*
	%	*<85*	*rel. humidity*
	K	*3*	*above dew point*
Consumption	*kg/m^2*	*~ 0,3*	*primer*
	kg/m^2	*~ 0,7*	*scratch and levelling coat*
	kg/m^2	*~ 0,9*	*for strewing layer*
	kg/m^2	*~ 1,2*	*quartz leveling layer*

Samples were made by following the instructions from the technical sheet. The epoxy resin was poured into molds so that prismatic samples 4x4x16 cm were obtained. They were left to stand for two weeks at room temperature. Samples were made in the ratio 1: 5 = resin: hardener, as well as 1.5: 1 = MC DUR 1320 VK: quartz (0.1 - 0.3).

The test was performed on two types of samples: pure epoxy samples, and on samples with quartz filler. The experiment was done in two phases. The first phase was done with the 200 kN Amsler hydraulic press. Flexural strength and compressive strength were tested on three 4x4x16 cm prismatic specimens, which had previously hardened in the mold. It is important to note that the samples hardened for one month, instead of the prescribed one-week time. The reason for the longer hardening was the thickness of the prismatic sample of 4 cm, instead of the usual few millimeters, as much as this coating should be applied in practice, and the consequent slower hardening of inner material. The appearance of the samples and measurement of their weight are shown in Figure 1.

Figure 1. *Epoxy samples and the measurement of mass*

The results of testing of density, flexural and compressive strength are given in Table 4.

Table 4.Density, flexural and compressive strength of the tested epoxy resin

Density	*Mass (g)*	*Volume (cm^3)*	*Density (g/cm^3)*	*Average density (kg/m^3)*
	287,9	*254.24*	*1.132*	*1128*
	286,5	*249.6*	*1,148*	
	288,0	*260.76*	*1,105*	
Flexural strenght	*Ultimate load (kN)*		*Average (kN)*	*Average flexural strength (MPa)*
	10,1	*9,1*	*10,3*	*25,8*
	12,1	*10,3*		
	10,9	*9,4*		
Compresive strenght	*Ultimate load (kN)*		*Average (kN)*	*Average compressive strenght (MPa)*
	96	*101*	*98,7*	*61,7*
	95	*102*		
	94	*104*		

In the second phase, a "Pull-off" test, in order to obtain bond strength, was performed. The samples were made as a 2 mm cover over the C35/45 concrete plates, in two series – one with and the other without quartz filler. The epoxy cover is shown on Figure 4, and the results of the tests are given in the Table 5.

Figure 2. *Surface with, and without quartz filler (left) and the apparatus used (right)*

Table 5.The obtained bond strength results

	Pure epoxy	***Comparison***	***Epoxy with quartz filler***	***Fracture***
1.	*7,54 kN – 3,84 MPa*	>	*6,57 kN – 3,34 MPa*	*Fracture of the concrete*
2.	*7,28 kN – 3,71 MPa*	<	*9,33 kN – 4,57 MPa*	*Fracture of the concrete*
3.	*8,76 kN – 4,46 MPa*	>	*7,72 kN – 3,93 MPa*	*Fracture of the concrete*

3. DISCUSSION

The obtained values of density ranged between 1105 kg/m^3 and 1148 kg/m^3, with the average of 1128 kg/m^3. Generally, these values are expectedly lower than the values for mortar and concrete, and lower than the values given in the technical data sheet. The values of flexural and compressive strengths (25,8 MPa and 61,7 MPa, respectively), obtained on 4x4x16 cm prisms (Fig. 5) are uniform and moderately high. In comparison to the cement composites, and having in mind ranges of the values expected for epoxy resins, it can be noted that they have reached satisfactory strengths.

Figure 3. *Compressive strength test using Amsler hydraulic press*

The pull-off strengths were obtained in the second phase. On the basis of the results that ranged between 3,71 and 4,46 MPa for the epoxy with quartz, and between 3,34 and 4,57 MPa for pure epoxy without quartz, the observation is that very similar bond strengths were obtained.

4. CONCLUSIONS

Although not a relatively new material, and although it has various applications, epoxy is not used as often, compared to other competing materials. The most likely reason for this problem is their price. In the case of industrial floors, investors mostly lean towards the cheaper solutions, without considering the long-term benefits of such floors. Therefore, besides the importance to perform according to the instructions, regulations and correctly, laboratory tests can help to confirm the results, and to opt the performances.

Testing of epoxy, normally used as a coating for industrial floors, was done on prismatic samples measuring 4x4x16 cm, and without any filler. After a prolonged curing period, density of 1128 kg/m3 was obtained, while the flexural and compressive strength reached 25,8 MPa and 61,7 MPa on average, respectively. The curing period had to be longer because the prescribed thickness for this material is only a few millimeters. Based on the investigated physical and mechanical properties of epoxy, it could be concluded that the use of this type of epoxy showed lower density and competitive values of compressive and flexural strength, meaning that high mechanical properties can be achieved even for lower density of the material. On the basis of bond strength test, it could be concluded that quartz filler slightly contributes to the improvement of strength, compared to pure epoxy sample. This effect was most likely due to the different procedure of the epoxy casting, than the one practiced on industrial floors. Also, quartz filler would significantly improve the anti-slip properties of epoxy coatings, but this test was not conducted. Regarding pot-life, a conclusion can be made that it was acceptable, and didn’t induce additional time dependent stresses on the process of casting.

Future developments in epoxy formulations intended for parking lots and industrial floors include: price reduction, automated production of such floors (typical sizes of floors 1000-3000 m^2), increased speed of placement, as well as changing the consciousness of future users due to their long-term advantages and possibilities. However, there are certain limitations in the use of these resins, such as their sensitivity to water and toxicity. Also, in order to ensure that epoxy floors maintain their high application rate and to be present in the industrial flooring market, improvements will need to be made in the following areas:

- Sand-filled floors often wear excessively if they are subjected to wet trucking, probably owing to sand worn from the surface forming a „grinding paste“ and causing rapid wear.
- For the efficient laying of trowelled floors, skilled labour is required. A mechanical means of laying would speed up the process and decrease the necessity for skilled manual trowelling.
- Epoxy floors will need to be made resistant to steam cleaning. At present they are failing, possibly due to the difference in the coefficients of thermal expansion of the floor and sub-floor concrete, or to the increase in the vapour pressure of the moisture held in the concrete sub-floor.

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