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New solvent-free epoxy dispersion in building practice

1. Introduction

Epoxy resins constitute an important group of resins which have been launched on the market immediately after WWII by the Ciba Company and Czechoslovakia was the second country in Europe that started the industrial production of epoxides (1). First applications of epoxy resins were in electro-insulating paints and dental technology. Gradually, epoxy dispersions started to be applied in the building industry, from coating compositions (2, 3) through moulding materials, adhesives, mastics, reinforced plastics, floor coverings, casting resins, etc. (4). Epoxy dispersions belonging to the sphere of waterborne substances were put on the market in the 1980s and 1990s (5). Although belonging to relatively new products, they have undergone a certain development during their short existence (6, 7, 8). **Type I dispersions** (1960) were based on waterborne low-molecular resins which had a number of disadvantages, among others, high brittleness. **Type II dispersions** (1980) were based on the dispersion of medium- or high-molecular epoxy resin in water. **Type III dispersions** are epoxy dispersions containing either no auxiliary organic solvents or only a little amount. These types of dispersions were first mentioned in the patent literature approximately 15 years ago, and they have, together with the „high solids“ systems and powdered or radiation-hardened binders, gradually been included in the so-called „eco-friendly coatings“, i.e. ecologically friendly binders (3).

The Research Institute for Synthetic Resins, SYNPO, a. s., Pardubice, Czech Republic, developed in 2006-2010, within the project of the Ministry of Industry and Trade, No. FT-TA 3/056, type III epoxy dispersion labeled CHS Epoxy 160V55, provisional name E1-M, including the aminic hardener of Telalit 1040 (8). The possibilities of application of epoxy dispersions in the building industry and their subsequent physical-mechanical and durability tests by the dispersions of additive materials were carried out by the Department of Building Testing, Faculty of Civil Engineering, BUT in Brno, Czech Republic. With varied success, we tested the use of E1-M dispersion in the modification of ordinary concretes (6), in the protection of concrete surface from the adverse effects of acid gases, and in the improvement of properties of concrete recyclate and concrete made from concrete recyclate (9, 10, 11). This epoxy dispersion will certainly find application in the building practice in spite of the comparatively high price of polymer inputs.

The aim of this research is a) to study the effect of waterborne epoxy dispersions on mechanical properties of concrete , b) the effect of coating on permeability of acid gases, and c) to compare the adhesive strength of a modern waterborne solvent-free dispersion adhesive with the relatively long established and practice-tested ceramic and classic solvent-based epoxy adhesives in gluing concrete, precise brick blocks, porous concrete or cladding elements. The advantage of waterborne dispersion adhesives is their trouble-free application even on a relatively humid adherend, which enables their wide use in the sphere of hydraulic structures, in refurbishments, maintenance of buildings and any other work with humid substrate. It would be beneficial to find such applications for the solvent-free epoxy dispersion where the advantages (adhesion to wet surfaces, harmlessness for health, odourlessness, high strength, chemical resistance after hardening) would exceed its relatively high price. On the other hand, the search for applications is limited especially by the aqueous character of the dispersion E1-M (water content approx. 50%), which requires temperatures above the freezing point (technological and storing), and by the absorption capacity of the connected or protected adherend or substrate. No less important condition for the applicability of water dispersions is the non-greasiness of the surface, which is definitely not fulfilled by either thermoplastics or thermosets.

2. Experimental

2.1. Epoxy dispersions added to the green concrete

Three types of polymers (resin E 200V55 + hardener Telalit 180; resin E160V55 (E1-M) + Telalit 180; resin E 160V55 (E1-M) + Telalit 180 + diisobutyrate D400) were used as additions into two formulations of cement concrete (see Table 1) to increase tensile strength and adhesion to the base. Diisobutyrate D400 is a substance that decreases evaporation of water from the dispersion and thus protects it from cracking during drying. Polymer dispersions, solutions of soluble monomers and polymers, resin emulsions or liquid resins without diluents which are not sensitive to humidity are used as polymer additions. Dispersions from thermoplastic and elastomers are used for polymer-cement composites production. From commonly used dispersions, the polyvinyl-acetate, vinyl-acrylic, styrene-acrylic and acrylic dispersions (12) can be

Table 1

MORTAR MIXTURE FORMULATIONS FOR MAKING STANDARD BEAMS 40x40x160 mm, tESTS OF MECHANICAL PROPERTIES OF PCC

Formulation	Estimated comp. strength in 28 days, MPa	Actual comp. strength in 28 day, MPa	Amount of fine aggregate, g	Amount of cement CEMI 42.5R, g	Amount of batch of water, l	Amount of emulsion in wt. of cement, %	Sort of emulsion
I	16.0	9.0	1580.0	153.6	0.107	0	without
		7.0				3	200V55+Telalit 180 E1-M+Telalit 180 E1-M +T180/D400
		14.0				6	
II	25.0	19.0	1529.0	202.0	0.112	0	without
		23.6				3	200V55+Telalit 180 E1-M+Telalit 180 E1-M +T180/D400
		13.0				6	

taken into account. From the in-water soluble resins, for example melanin resin or furyl alcohol can be used. Epoxy dispersions were not commonly used so far.

Optimization of the amount of polymer additions is one of the goals of this work. Generally, value 5-10 % of the weight of cement is given in the literature (13). Composite resistance against degradation effects of permanent placement in the humidity and water is threatened by bigger amount of the polymer additions.

From the technological point of view the polymer-cement mixture production is similar as production of common cement-concrete mixtures. Polymer dispersion must be admixed to the batch water. The most of dispersions have plastification effect on the concrete mixture. Moreover, foam is made from the dispersion, therefore antifoaming substances must be added into polymer-cement mixtures.

2.2. Effect of coating on permeability of acid gases

The influence of the coating on permeability of aggressive CO_2 was determined on selected concrete samples which were fabricated by three different recipes with various values of compressive strength (see Table 2).

On the basis of mutual comparison of carbonated depths of samples with the coats and without them the effect of applied coats on carbonation depth can be assessed. Single-layer coatings were 60 μm thick, double-layer coatings then 120 μm. The drying interval in the case of double-layer coating was 24 hours at 20°C. The hardening time of coatings was one month. It can be done in the environment of concentrated 98% CO_2 and in laboratory atmosphere where there is about 0.03% CO_2 (14). Four combinations of waterborne dispersions and hardeners were applied separately on the concrete samples:

Coating A: 2 layers of dispersion 200V55 + hardener Telalit 180

Coating B: 2 layers of dispersion DOW XZ 92533 + hardener XZ 9241.01

Coating C: 1 layer of dispersion E 200V55 + hardener Telalit 180 and 1 layer of dispersion E1-M + hardener Epostyl 217 V

Coating D: 2 layers of dispersion E1-M + hardener Epostyl 217 V

The necessary data about the resins and additives used:

Resin 1:

CHS Epoxy 200V55 is a water dispersion of a medium molecular weight epoxy resin. The system comprises of a modified epoxy resin and a special hardener Telalit 180. Viscosity (25°C): 0.1-0.7 Pa.s DIN 53015. Content of non-volatile matters (2 h/140°C): 54-58 wt.% in accordance with ISO 3251.

Hardener 1:

Telalit 180 is a universal hardener for CHS Epoxy waterborne epoxy systems. Telalit 180 is a water-based solution of an epoxy-polyamine adduct. Viscosity (25°C): 0.75-1.1 Pa.s DIN 53015. Recommended mixing ratio CHS Epoxy 200 V 55 : Telalit 180 - 100pbw. : 27pbw.

Epostyl 217 coating system:

It is a two-component system based on the dispersion, CHS Epoxy 160V55 (waterborne solvent-free solid epoxy dispersion) and hardener. Mixing ratio 1 : 1.

Resin 3:

DOW XZ 92 533 is a waterborne epoxy dispersion from DOW Company. Viscosity (23°C): 0.497 Pa.s ČSN 64 0349. Non-volatile (2h/130°C): 46.7 wt.% in accordance with ČSN EN ISO 3251.

Table 2

MORTAR MIXTURE FORMULATIONS FOR MAKING STANDARD BEAMS 40X40X160 MM, TESTS OF POLYMER COATINGS ON HARDENED MORTAR

Formulation	Estimated comp. strength 28 days, MPa	Actual comp. strength 28 days, MPa	Amount of fine aggregate, g	Amount of cement CEMI 42.5R, g	Amount of batch of water, ml	Number of samples, piece
1	15.0	11.0	1600.0	200.0	185.0	30
2	25.0	24.0	1475.0	325.0	200.0	30
3	35.0	37.0	1350.0	450.0	212.0	32

Table 3

OVERVIEW OF ADHESIVES AND PENETRATIONS USED IN THE PULL-OFF TEST

Specimen Basic type	Type of adhesive and penetration	Type of hardener	Mixing proportion A:B:(water)	Purpose in adhesion test
I. Dispersion	Adhesive L1 part A	Adhesive L1 part B	100:7	Tested adhesive
II. Ceramic	Adhesive Flexkleber elastic	-	5:1.4	Reference adhesive No.1
III. Epoxy	Adhesive E520 part A (resin)	Telalit 2007	2:1	Reference adhesive No.2
IV. Dispersion	Penetration CHS Epoxy 160V55	Telalit 1040	100:9:(109)	Penetrating system
V. Epoxy	Discs adhesive A	Discs adhesive B	2:1	Discs adhesive

Hardener 3:

XZ 92 441.01 from DOW Chemicals Ltd. Recommended mixing ratio DOW XZ 92 533 : XZ 92 441.01 - 100 pbw. : 20 pbw.

Resin E1-M (CHS Epoxy 160V55) is a tested dispersion, identification data cannot be published

2.3. Application of epoxy dispersions in the form of adhesives

The value which will help us to objectively determine the differences between adhesives as regards the quality of adhesion and cohesion is the maximum tensile strength caused by load acting perpendicularly on the surface of the applied adhesive expressed as adhesive strength in N/mm^2. The essentials of this test are stipulated by ČSN EN 1015 – 12. Although intended for testing mortars for masonry, this test will fully satisfy our need for a comparison of properties of the tested adhesives.

Using a device suitable for cutting concrete (core drilling machine), we will cut a circular surface with a diameter of 50mm. The area delimited according to the test specification must be treated by grinding, wetting or impregnation. Then we will apply adhesive (see Fig. 1) which is to be tested onto it and let it harden for 28 days (this time of hardening was the same for all adhesives used (also pure resin and ceramic) because of the subsequent comparison of adhesiveness). After hardening we will apply epoxy or acrylate resin (high-strength adhesive) on the layer of the tested adhesive and attach onto it a circular disc (pull-head plate) made of corrosion-resistant steel with a diameter of 50 ± 0.1mm and a minimum width of 10mm, with a central spike for attaching a Dyna Z 15 testing apparatus made by PROCEQ (see Fig. 2). After the adhesive hardens, we will attach the spike to the testing apparatus and generate tensile strength perpendicularly on the tested area. The velocity must be chosen so that the rupture occurs between 20 to 60 s. We record the test results and, if necessary, exclude all the results where the rupture occurred in the layer of the auxiliary (disc adhesive) adhesive between the circular disc and tested adhesive. The valid test result can have three forms of rupture:

a) rupture occurred between the adherend and adhesive, the adhesive has low adhesion to the glued material
b) specimen damaged in the layer of the tested adhesive, the cohesive strength of adhesive is lower than the cohesive strength of the adherend
c) specimen damaged in the adherend, which is the most favourable situation in testing adhesives, the adhesion and cohesion of adhesive is higher than the cohesive strength of the glued material.

The aim of this research is to compare a modern two-part waterborne dispersion adhesive marked L1 (the base of L1 is also type III epoxy dispersion labeled CHS Epoxy 160V55, provisional name E1-M) made by Synpo Pardubice with two types of reference adhesives, namely the silicate adhesive Flexkleber made by Knauf and the epoxy dispersion E520 made again by SYNPO Pardubice (15). The tested modern solvent-free two-part adhesive L1 consists of:

part A: 73% dispersion CHS Epoxy 160V55, 20% Kalcimat KO – 1/30, 0.5% Bentone LT, 0.2% Bayferrox 316

part B (hardener): 6.5% Telalit 1040

Table 3 gives an overview of the adhesives, penetrations and hardeners used in the pull-off test , their mixing ratios and their importance for testing.

The adhesion test was carried out on three types of substrates (adherends) which are common in the building practice. A detailed

Table 4

OVERVIEW OF ALL TYPES OF TREATMENT OF ADHERENDS IN RELATION TO THE USED TYPE OF ADHESIVE I., II. AND III

Specimen Basic type	Adherend	Treatment of adherend: Grinding of surface	Treatment of adherend: Penetration with dispersion IV.
I. Dispersion (tested adhesive)	concrete	yes	yes
			no
		no	yes
			no
	silicate	yes	yes
			no
	porous concrete	no	yes
II. Ceramic (reference adhesive 1)	concrete	yes	no
		no	
	silicate	yes	
	porous concrete	no	
III. Epoxy (reference adhesive 2)	concrete	yes	no
		no	
	silicate	yes	
	porous concrete	no	

Table 5

PHYSICAL-MECHANICAL PROPERTIES OF COMMON ADHERENDS

Adherend	Volume mass, kg/m^3	Apparent porosity, %	Compressive strength, MPa	Tensile strength, MPa
Concrete	2360.0	8.0	36.0	2.6
Silicate	1840.0	18.0	22.0	1.6
Porous concrete	500.0	44.0	4.2	0.4

Fig. 1. Application of adhesive L1 on ground concrete substrate

Fig. 2. Testing apparatus Dyna Z 15 made by PROCEQ Company

overview of the adherends and their treatment before gluing is given in Table 4. Table 5 contains the physical-mechanical properties of adherends influencing the necessity of penetration and adhesiveness of the tested and reference adhesives.

The testing of the adhesive strength of the L1 adhesive on the porous concrete was complicated by a considerably high absorption capacity of the material (see Table 5 and Fig. 3). In the building practice, some surfaces are damped before the application of another contact layer. The prerequisite for further testing would therefore be that, if the surface of porous concrete is moistened, it will not remove water from the tested adhesive L1. The absorption capacity of porous concrete, however, is very high, the hardened layer of adhesive developed cracks although the tested surface was considerably soaked with water. The disintegration was not as high as in the previous case (see Fig. 4) but the result still has not been in accordance with the required properties. That is why we decided to penetrate the surface before the application of the adhesive itself (16). The dispersion used was CHS Epoxy 160V55 with a higher content of water mixed in the system than in the case of adhesive L1. The application of penetration came up to expectations and the applied adhesive L1 created a smooth compact surface without cracks after harding (see Fig. 5).

3. Results and discussions

3.1. Epoxy dispersions added to the green concrete

The research aim of the paper is to compare the effect of water-based epoxy dispersion on mechanical properties of polymer-cement concrete (PCC) or mortar. Domestic older solvent (E 200V55) or newer solvent-free (E 160V55, provisionally E1-M) dispersions were used. Concrete was prepared from two recipes. When using formulation I, the compressive strength is commonly up to 16 MPa, with formulation II up to 25 MPa, in both formulations we used portland cement CEM I 42.5 R (see Table 1). Results of tensile bending test and test in compression according to the concentration of dispersions (0.3 and 6 wt.% of cement) are in Figure 6a and Figure 6b. Polymer-cement concrete (mortar) prisms of dimensions 40x40x160 mm were tested first in three-point bending test, their fragments in compressive test.

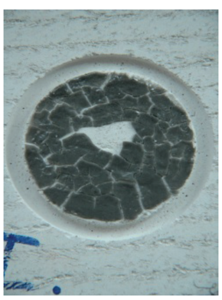

Fig. 3. The surface of hardened but over-dried adhesive L1 without penetration and substrate moistening

Fig. 4. The surface of hardened adhesive L1 with moistening but without substrate penetration

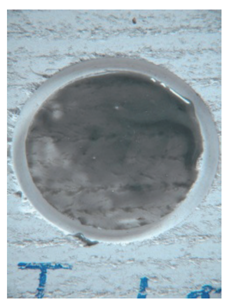

Fig. 5. The surface of hardened adhesive L1 with substrate penetration

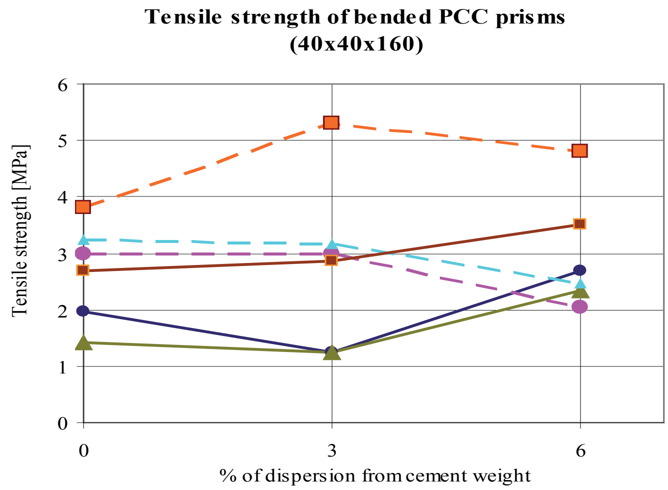

Fig. 6a. Tensile strength of bended PCC prisms (40x40x160)

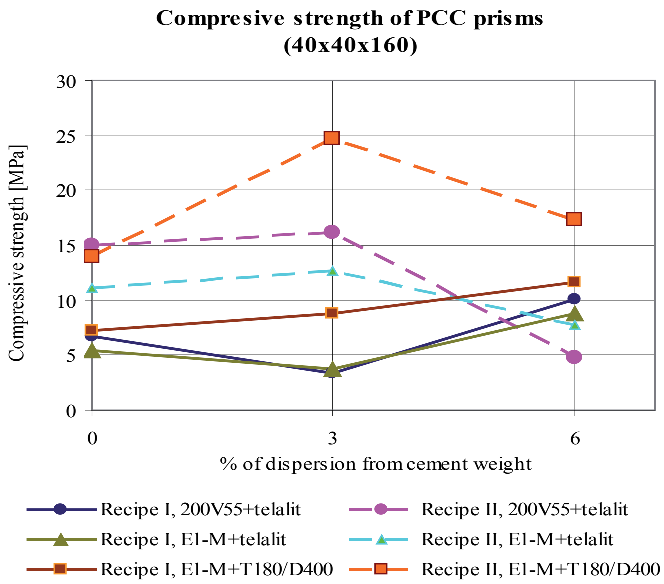

Fig. 6b. Compressive strength of PCC prisms' fragments

3.2. Effect of coating on permeability of acid gases

Carbonation depth of samples placed in 98% CO_2 environment is compared to the depth of natural carbonation in laboratory environment. The depth of carbonation had been monitored for six months. Method of assessment is shown on fragments of concrete (mortar) prisms, Fig. 7 and 8. Fig. 7 is a result of starting test at the beginning of testing period (half year). Three reference specimens are in Fig. 7 on the left. They did not have any protective cover before the test. Then follow specimens A, B, C and D covered by the coat of the same named combination of dispersion and hardener. The sample on the right was not tested (sprayed by phenolphthalein) and is shown because of the comparison.

3.3. Application of epoxy dispersions in the form of adhesives

An overview of the results of determined adhesive strengths of the tested and reference adhesives on three variants of surface treated materials is given in graphs in Fig. 9, 10 and 11.

Fig. 7. Concrete specimens after starting assessment on CO_2 penetration

Fig. 8. Concrete specimens after half year acting of CO_2

In the case of adhesive L1, during the adhesive strength test, the material of adherend was pulled off, i.e. adhesion on material and internal cohesion of the adhesive L1 is higher than the cohesion of the glued material (see Fig. 13, 14). The use of penetration improves the cohesion proper of the silicate adherend, in the case of porous concrete it slows down the drying of applied dispersion adhesive. It is interesting to compare adhesive L1 with type II reference adhesive, i.e. the ceramic binder Flexkleber made by Knauf, where in the case of glueing on concrete and silicate body (see Fig. 12) there was a rupture in the layer of the adhesive itself – it was therefore a failure of the internal cohesion of the adhesive.

In the adhesive strength test of the adhesive on a specimen of concrete, all the three types of adhesives complied, the tensile strength of the surface layers of porous concrete was exceeded by the both adhesive and cohesive strengths of the tested adhesives. The best was, however, the classic solvent-based adhesive III, i.e. Epoxy E520.

4. Conclusion

Modern waterborne epoxy dispersions with a zero content of VOC are high-quality ecological materials, commonly used nowadays

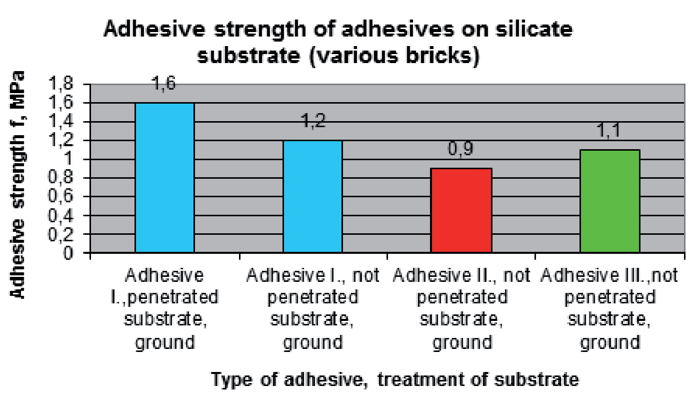

Fig. 10. Adhesive strength on silicate substrate

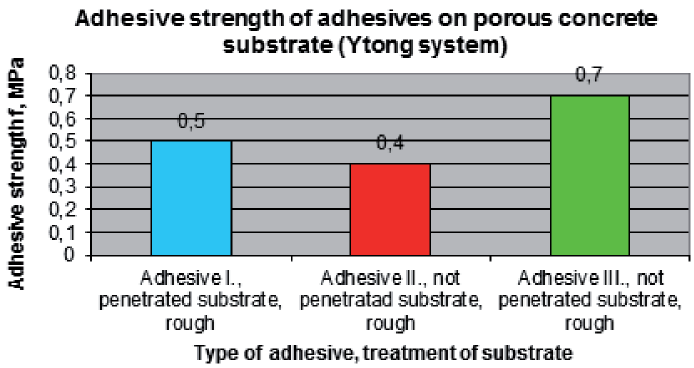

Fig. 11. Adhesive strength on porous concrete substrate

it is necessary to consider the benefit of a higher adhesive strength versus price of the dispersion adhesive (min. 8€ / 1 litre of L1) or the necessity of surface treatment by grinding and penetration.

By way of conclusion it is possible to say that the tested two-part dispersion building adhesive L1 (type I) came up to expectations and can therefore be recommended for building purposes, i.e. especially for executing contact joints of moisture-absorbent materials, such as brick - brick, concrete - concrete, concrete - brick, porous concrete - concrete, etc. at temperatures above freezing point. It is supposable that joints of similar quality can also be achieved by gluing other pairs of materials, at least one of which will be moisture-absorbent.

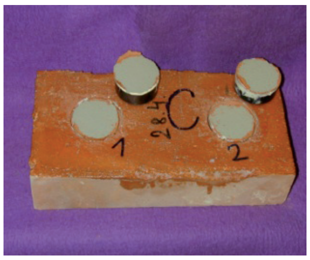

Fig. 12. Cohesive failure of Flexkleber adhesive

Fig. 13. Cohesive failure of silicate adherend

Fig. 14. Cohesive failure of concrete adherend

for treatment of wood, metals and glass. The applied research at the Faculty of Civil Engineering of BUT brought these materials closer also to wider building practice, whether it be the additivation of cement concretes, protective coatings of ceramic structures or durability improvements of concretes made from recycled concrete. However, due to its relatively high price, the largest volume of epoxy dispersions will probably head towards special applications in future, e.g. maintenance, dry masonry and gluing.

Concretes of two different recipes were prepared, 3 and 6% of epoxy dispersion was added. For comparison also specimens without any dispersion were tested. Tensile strength in bending and compressive strength were tested by all samples. The best results (by tensile bending tests or compressive tests) were achieved in the concrete of higher strength which contained 3% of dispersion E1-M with hardener Telalit 180. By the concentration 6% of dispersion the strengths decreased, especially in case of compressive strengths and concretes of the second recipe. Both mechanical properties are little bit improved when adding 6% of epoxy dispersion to concrete of lower strengths (recipe I).

Following conclusions can be determined on the basis of tests of four different types (A, B, C, D) of water-based epoxy emulsions according to penetration of CO_2 during half-year accelerated test:

– the highest-level protection of cement mortar had solvent epoxy emulsion type A and also combination of solvent + solvent-free type (C) where the first coat was made as in type A combination
– on the other hand, foreign emulsion of type B hadn't protected cement mortar during 6 months acting of aggressive CO_2

Ceramic adhesives, frequently used nowadays, can be successfully replaced with waterborne epoxy dispersions but in some situations

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