Preparation and Properties of Epoxy Adhesives Blends

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Abstract

This research aims to improve the cohesion forces , thermal and mechanical properties and reducing cost for epomort epoxy adhesive by addition tarcoat epoxy adhesive as blends with ratio (25 , 50 , 75 V %) . The results of bending strength testing , compression strength testing and shore A hardness increasing for 75 % epomort + 25 % tarcoat epoxy adhesive blends more than 100 % epomort , 50 % epomort , 25 % epomort and 100 % tarcoat epoxy adhesive blends .The results of adhesion strength testing between concrete – epoxy adhesive blends at 25 °C and -2 °C show the failure has taken place in concrete , while at 90 °C the failure has taken place in concrete for 75 – 50 – 25 % epomort and 100 % tarcoat epoxy adhesive blends except 100% epomort epoxy adhesive the failure has taken place in epoxy adhesive . The crystallization temperature ($T_{\rm c}$) , melting temperature ($T_{\rm m}$) and decomposition temperature ($T_{\rm d}$) of all ratio epoxy adhesive blends are converged or nearby . Reducing cost of epomort epoxy adhesive with addition tarcoat epoxy adhesive as blends by ratio (25 , 50 and 75 %) . The 75 % epomort + 25 % tarcoat epoxy blend gives better mechanical properties , thermal properties and low cost .

Key words: epoxy adhesive , adhesion strength testing , thermal analysis , mechanical properties , epoxy blends .

الخلاصة

الكلمات الدالة : لاصق ايبوكسي , مقاومة التلاصق , التحليل الحراري , الخواص الميكانيكية و مزيج ايبوكسي . محمة محمد

1. Introduction

For more than 30 years, epoxies have played a critical role in modren industries. They were first introduced into the hybrid industry and enjoyed much success in bonding components, protecting devices, and hermetically sealing packages [Pecht, M. G., Nguyen, L. T., and Hakim, E. B., 1995]. Epoxy resins are widely used as adhesives, coatings, paints, composites, and dental therapy, because they have good adhesion, little dimensional changes, remarkable chemical resistance, and mechanical properties [Schmidt, R. G.; Bell, J. P., 1986, Kinjo, N.; Ogata, M.; Nishi, K.; Kaneda, A., 1989, Gaschke, M., Dreher, B. J., 1976, Baley, C., Davies, P., Grohens, Y., Dolto, G., 2004 and Fujihara, K., Teo, K., Gopal, R., Loh, P. L., Ganesh, V. K., Ramakrishna, S., Foong, K. W. C., Chew, C. L., 2004].

The blending of existent polymers to create new materials with enhanced properties has become a successful alternative to the expensive synthesis of completely new polymers. The possible combinations of polymers seem endless, and for the past 30 years, research has resulted in a large number of commercial applications.

[Bucknall C. and Partridge I., 1983, Huang M., Zhu K., Pearce E. and Kwei T., 1993 and Gandini A., Allen G., 1992].

In a previous study, the researchers used two novel hyperbranched polymer (HBP)/epoxy blends were evaluated to determine their efficiency as blend modifiers and tougheners in a brittle epoxy resin [Thomas J. Mulkern, Donovan Harris and Alan R. Teets, 1999]. Other researchers used fire behavior of the BPC cyanateepoxy blends was studied in flaming and nonflaming combustion, using fire calorimetry and pyrolysis-combustion flow calorimetry (PCFC), respectively [Richard N. Walter, 2002]. And other researchers used the epoxy / phenolic resin blends were formulated by a sequential mixing process. The thermal conductivity (k) of the blends has been measured as a function of temperature over the range 303-373K°. The results show that the values of k increase with increasing Phenolic weight fraction. It also considers an increase in the effective thermal conductivity at high temperatures due to increasing the intermolecular vibrations. [Sammani Ali S. and Nayef Saleh A., 2004]. Other researchers used investigate the performance potential of cyanate ester / epoxy blends at higher fluence levels without significant degradation of their mechanical properties [R. Prokopec and Others, 2008]. other researchers used Radiation curing of an epoxy resin / thermoplastic blend is carried out in order to produce polymer matrices for high-performance composites. A suitable choice of both curing process and formulation parameters allows to irradiate at about 60°C. Moreover hydrothermal ageing is performed on cured materials and their properties have been evaluated [S. Alessi and Others, 2009].

This study aims to improve the cohesion forces , thermal and mechanical properties and reducing cost for epomort epoxy adhesive by addition tarcoat as blends with ratio 25 , 50 and 75 % .

2. Materials and Methods

Epomort (Epomort 1000) Epoxy Adhesive

It is a 100% reactive, 2 component material designed as a moisture insensitive adhesive and binder for numerous application needs. Epomort provides normal working time when bonding dry or damp concrete at temperature above $4^{\circ}C$.

Mixing - All materials should be in the proper temperature range of 16° C - 32° C. Mix part A and part B (resin & hardener) for 2 minutes . For ease of mixing, add the part B to part A (not the reverse). The epoxy must be well mixed to ensure proper chemical reaction according to CMCI company . (The price of epomort epoxy adhesive is (25\$) per liter in the domestic markets).

Tarcoat Epoxy Adhesive

It is a solvent free two component high build amine-cured coal tar modified epoxy coating. It has excellent adhesion with steel surfaces.

<u>**Mixing</u>** - Add Part B, the hardener into the Part A . Mix for at least 3 minutes till uniform consistency is obtained according to CMCI company . The price of tarcoat epoxy adhesive is (5\$) per liter in the domestic markets .</u>

Preparation of Blends

Table (1) show ratio of mixing for epomort – tarcoat epoxy adhesive blends . Shore A hardness and compressive properties were evaluated in accordance with ASTM D 2240 and D 786 standards, respectively. In addition, the pull off adhesion strength was determined according to ASTM D 7234 standards . The flexural properties were evaluated in accordance with ASTM D790M standards . The melting and glass transition temperature of the blends was determined by using DSC . The conditions used were: heating rate $10^{\circ}C/min$ for $400^{\circ}C$.

Surface Preparation

New concrete must be a minimum of 28 days old and possess an open, porous and textured surface with all curing compounds and sealers removed. Old concrete must be clean and well textured. All oil, dirt, debris, paint and unsound concrete must be removed. The surface should be prepared mechanically using abrasion machine or sandblast. The final step in cleaning should be the complete removal of all residue with a vacuum cleaner or pressure washer. Acid etching is acceptable only when mechanical preparation is impractical. It is recommended that only contractors experienced in the acid etching process use this means of surface preparation. Allow the concrete to completely dry. Figure (1) (a, b) Appearance of samples of concrete adhesion strength testing.

Environmental Testing

Samples of concrete adhesion strength testing put under some environmental condition such as :

- 1- At (25 °C).
- 2- At $(90 \,{}^{\circ}C)$ for $(4 \, hr)$.
- 3- At $(-2^{\circ}C)$ for (4 hr).

Samples preparation for thermal analysis

Samples of (epomort – tarcoat) epoxy adhesive blends were cutting by using a sharp knife. Two pieces were placed in aluminum crucible so that the flat surfaces rested face-down on the bottom. This ensured good thermal contact between the crucible and the sample. The sample mass was 10-15 mg. The crucible was sealed using a lid with a hole , according to ASTM D 3418-03 and ASTM E 1356-03 [ASTM D 3418-03, 2004 and ASTM E 1356-03, 2003].

According to the standard, it is important that the sample does not move during the measurement. This was done by placing the crucible on a flat steel surface and pressing the lid onto the sample using the flat end of a rod.

Differential Scanning Calorimetry (DSC) Testing

The measurements were performed using a SHIMADZU DSC-60 made in Japan 2010. The temperature program used ASTM D 3418-03 and ASTM E 1356-03 [ASTM D 3418-03, 2004 and ASTM E 1356-03, 2003].:

- Start temperature: At 25 °C
- End temperature: 400 °C
- Heating rate: 10 °C/min

3. Results and Discussion

Adhesion Strength Testing

Figures (2 and 3) summarizes the results of adhesion strength testing between concrete – epoxy adhesive blends at 25 °C and -2 °C respectively, the results show the failure has taken place in concrete cohesion failure (CCF) but don't happened in epoxy adhesive blends. Because the adhesion forces between concrete and epoxy adhesive blends and adhesive cohesion forces more than concrete cohesion forces, result for mechanical interlocking theory and adsorption theory between concrete and epoxy adhesive blends , and electrostatic attraction theory between adhesive chains are very good, the bond depend on adsorption theory, adhesive molecules adsorption inside concrete surface and formation bonds between surface for concrete – epoxy adhesive blends good adhesion force . For that the failure happened in concrete zone. [Petrie, Edward , M, 2007]

Figure (4) summarizes the results of adhesion strength testing between concrete– epoxy adhesive blends at 90 °C , the results show the failure happened in concrete concrete cohesion failure (CCF) for B , C , D and E adhesive blends except A adhesive the failure happened in epoxy adhesive blends adhesive cohesion failure (ACF) .Because of concrete cohesion forces (CCF) less than adhesive blend cohesion forces (ACF) and adhesive blend adhesion forces between concrete – epoxy adhesive blends for B , C , D and E adhesive blends , same reason up in figure (2,3) . About A adhesive the glass transition temperature (Tg = 89 °C) according to DSC as figure (8) , the treatment at 90 °C above Tg for A adhesive for that A adhesive became soft and elastic for that A adhesive cohesion forces (ACF) at 90 °C less than (concrete cohesion forces (CCF) and adhesion forces between concrete – epoxy adhesive blends [Petrie, Edward , M , 2007 and Raymond A. Higgins , 2006] .

Mechanical Properites

Figures (5 and 7) summarize the results of bending strength testing , compression strength testing and shore A hardness respectively , the results give increasing in compression strength , bending strength and shore A hardness for B adhesive blends more than A, C, D and E adhesive blends .

Because of increasing mechanical interlocking between B adhesive blends chains resulting for increasing cross linking (curing) between B adhesive blends chains , in figure (9) according to DSC area under curve of crystallization reduce by increase ratio of cross linking , for that area under curve of crystallization for B adhesive blends less than area under curve of crystallization for A , C , D and E adhesive blends for that the results give increasing in compression strength , bending strength and shore A hardness for B adhesive blends more than A , C , D and E adhesive blends [William D. Callister and Jr. , 2000 and Hal F. Brinson and L. Catherine Brinson , 2008] .

Thermal Analysis Testing

The main features of the DSC analysis of various blend compositions are summarized in Table (2).

Figures (8-12) summarize the results of thermal analysis of DSC curve for A, B, C,D and E adhesive blends . Appearance of results of DSC curves crystallization temperature (T_c), melting temperature (T_m) and decomposition temperature (T_d) of all ratio epoxy adhesive blends are converged or near . Area under curve of crystallization for B adhesive blends less than area under curve of crystallization for A, C, D and E adhesive blends .

Because of the types of epomort and tarcoat are epoxy adhesive and it can mixing with easy because it is miscible blend between two constituents for that crystallization temperature $(T_{\rm C})$, melting temperature $(T_{\rm m})$ and decomposition temperature (T_d) of all ratio epoxy adhesive blends are converged or nearby [William D. Callister and Jr. , 2000 and Hal F. Brinson and L. Catherine Brinson , 2008].

Reducing of cost Testing

Reducing of cost for Epomort epoxy adhesive by ratio 20, 40 and 60 % respectively when addition Tarcoat epoxy adhesive as blends by ratio 25,50 and 75 % respectively. Table (3) shows ratio of cost reducing for epomort – tarcoat epoxy adhesive blends .

4. Conclusions

1- The results of adhesion strength testing between concrete – epoxy adhesive blends at 25 °C and -2 °C show the failure happened in concrete, while at 90 °C the failure happened in concrete for B, C, D and E adhesive blends except A adhesive the failure has taken place in epoxy adhesive blends.

- 2- The results of bending strength testing , compression strength testing and shore A hardness increasing for B adhesive blends more than A , C , D and E adhesive blends.
- 3- Appearance of results of DSC curves the crystallization temperature (T_C) , melting temperature (T_m) and decomposition temperature (T_d) of all ratio epoxy adhesive blends are converged or nearby.
- 4- Reducing of cost for epomort epoxy adhesive by ratio 20, 40 and 60 % respectively when addition tarcoat epoxy adhesive as blends by ratio 25, 50 and 75 % respectively.
- 5- The 75 % epomort + 25 % tarcoat epoxy blend gives better mechanical properties , thermal properties and low cost.

5. References

- ASTM D 3418-03, 2004, Standard Test Method for Transition Temperatures of Polymers by Differential Scanning Calorimetry.
- ASTM E 1356-03, 2003, Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry.
- Baley, C., Davies, P., Grohens, Y., Dolto, G., 2004, "Application Composite Material".
- Bucknall C. and Partridge I., 1983, "Polymer".
- Fujihara, K., Teo, K., Gopal, R., Loh, P. L., Ganesh, V. K., Ramakrishna, S., Foong, K. W. C., Chew, C. L., 2004, "Composite Science Technology".
- Gaschke, M., Dreher, B. J., 1976, "Coating Technology".
- Gandini A., Allen G., 1992, "Comprehensive Polymer Science; Polymers from Renewable Resources. Pergamon Press", New York.
- Huang M., Zhu K., Pearce E. and Kwei T., 1993, "J. Appl. Polym. Sci.".
- Hal F. Brinson and L. Catherine Brinson , 2008, "Polymer Engineering Science and Viscoelasticity An Introduction", USA .
- Kinjo, N.; Ogata, M.; Nishi, K.; Kaneda, A., 1989, "Advance Polymer Science ".
- Pecht, M. G., Nguyen, L. T., and Hakim, E. B., 1995 ," Plastic Encapsulated Microelectronics ", J Wiley, New York .
- Petrie, Edward , M , 2007 , " HANDBOOK OF ADHESIVES AND SEALANTS " , Second edition , USA .
- Raymond A. Higgins , 2006 , " MATERIALS FOR ENGINEERS AND TECHNICIANS " , $4^{\rm th}$ edition .
- Richard N. Walter, 2002, "Fire-Resistance Cyanate Ester Epoxy Blends ", US department of transportation.
- R. Prokopec and Others , 2008 , " Mechanical Behaviour of Cyanate Ester / Epoxy Blends after Reactor Irradiation to High Neutron Fluences " , Austria .
- Schmidt, R. G.; Bell, J. P., 1986, "Advance Polymer Science".
- Sammani Ali S. and Nayef Saleh A. , 2004 , " Thermal Properties of Epoxy (DGEBA) / Phenolic Resin (Novolac) Blends " , K.S.A. .
- S. Alessi and Others , 2009 , " Epoxy Resin Thermoplastic Blends Cured by Ionising Radiation . Structure / Properties Relationships ", Italy .
- Thomas J. Mulkern , Donovan Harris and Alan R. Teets , 1999 , " Epoxy Functionalized Hyberbranched polymer/Epoxy blends " , Army research laboratory .
- William D. Callister and Jr., 2000, "MATERIALS SCIENCE AND ENGINEERING AN INTRODUCTION ", 5th edition, Department of Metallurgical Engineering, University of Utah, U.S.A.

biends.								
Symbol of	Ratio of	Epomort epoxy adhesive (%	Tarcoat epoxy					
blend	blends (%	V)	adhesive (% V)					
	V)							
А	100:0	100	0					
В	75:25	75	25					
С	50:50	50	50					
D	25:75	25	75					
Е	0:100	0	100					

Table (1) shows ratio of mixing for epomort – tarcoat epoxy adhesive blends .

 Table (2) shows results of thermal analysis for (epomort + tarcoat) epoxy adhesive blends by using DSC .

Types of Epoxy	$T_{g}(^{o}C)$	$T_{\rm C}(^{\rm o}{\rm C})$	Area under Curve of	$T_m(^{\circ}C)$	$T_d(^{o}C)$
Blends	ç		Crystallization (mJ)		
100 % Epomort	89.1	303.8	902.57	376.37	394.63
75 % Epomort +	Not	334.42	98.9	346.18	347.15
25 % Tarcoat	observed				
50 % Epomort +	Not	296.03	117.33	341.78	393.36
50 % Tarcoat	observed				
25 % Epomort +	Not	310.06	231.1	337.82	378
75 % Tarcoat	observed				
100 % Tarcoat	Not	311.64	246.02	342.32	386.27
	observed				

Table (3) shows ratio of cost reducing for epomort – tarcoat epoxy adhesive blends .

Symbol of	Ratio of blends (% V)	Ratio of cost reducing	Cost
blend		(%)	(\$)
А	100 : 0	0	25
В	75:25	20	20
С	50 : 50	40	15
D	25:75	60	10
Е	0:100	0	5



Figure (1) (a, b) Samples appearance of concrete adhesion testing.



Figure (2) shows results of concrete adhesion testing for concrete samples with

(epomort - tarcoat) epoxy adhesive blends at 25 °C .



Figure (3) shows results of concrete adhesion testing for concrete samples with (epomort – tarcoat) epoxy adhesive blends at (- 2 °C for 4 hr).



Figure (4) shows results of concrete adhesion testing for concrete samples with (epomort – tarcoat) epoxy adhesive blends at (90 °C for 4 hr) .



Figure (5) shows results of bending strength testing for (epomort – tarcoat) epoxy adhesive blends at 25 °C.



Figure (6) shows results of compression strength testing for (epomort – tarcoat) epoxy adhesive blends at 25 °C.



Figure (7) shows results of Shore D hardness testing for (epomort – tarcoat) epoxy adhesive blends at 25 $^{\rm o}{\rm C}$.



Figure (8) shows results of thermal analysis of DSC curve for (100 % epomort).



Figure (9) shows results of thermal analysis of DSC curve for (75% epomort + 25% tarcoat) .



Figure (10) shows results of thermal analysis of DSC curve for (50% epomort + 50% tarcoat).



Figure (11) shows results of thermal analysis of DSC curve for (25% epomort + 75% tarcoat) .



Figure (9) shows results of thermal analysis of DSC curve for (100 % tarcoat).