An investigation on physical response of modified adhesives used in medical applications

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Abstract: Glues and adhesives are increasingly used in medical applications. They can be utilized in disposal medical devices, structural bonds, bone cement, prostheses and other applications. For improve the adhesion of these materials, the liquid Hycar modifier is added and mixed. However, despite liquid modifier might modify the adhesion properties of glues and adhesives, the physical properties of these materials might be deteriorated. In this paper the physical response i.e. thermal and mechanical behavior of the adhesive modified with liquid rubber has been investigated for medical applications. In addition, scanning electron microscopy (SEM) is utilized in this work for better understanding the behavior of adhesive.

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1. Introduction

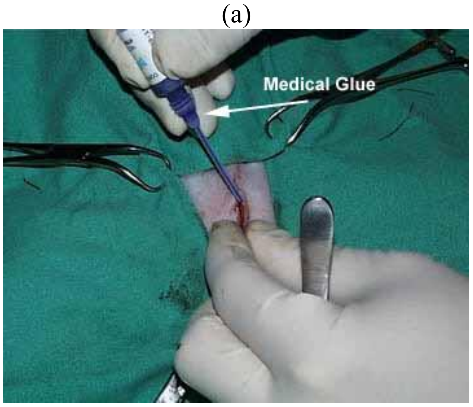
Recently, glues and adhesives are used in medical applications. Since the hardened, finished polymers are almost non-toxic, they can used in many medical applications such as disposal medical devices, structural bonds, bone cement, prostheses, etc.. In figure (1) some medical applications of the cut-cure adhesives e.g. stopping bleeding, surgical operations are demonstrated. Each medical adhesive has its own benefits and limitations that should be matched with its appropriate application. The primary types of adhesives used in medical device applications includes Acrylics, Epoxies and Styrene polymers. Challenges for medical devices that require skin contact can often be reduced to a wrestling match between adhesion and irritation.

Adhesion, the attachment between glue and substrate may occur either by mechanical means, in which the glue works its way into small pores of the substrate, or by one of several chemical mechanisms (Ratna *et al.* 2000). The strength of glue depends on many factors. In some cases, an actual chemical bond occurs between adhesive and substrate (Takemura *et al.* 1985). In others, electrostatic forces, as in static electricity, hold the substances together. A third mechanism involves the molecular forces that develop between molecules. A fourth means involves the moisture-aided diffusion of the glue into the substrate, followed by hardening (Takemura *et al.* 1985; Ratna *et al.* 2000).

Thermal stability is very important for High-temperature applications of an adhesive material. In order to use these materials in high temperature applications, the thermal properties of these materials should be evaluated (Takemura *et al.* 1985; Ratna *et al.* 2000; Ratna *et al.* 2001). In this work the Differential Scanning Calorimetry (DSC) is used to measure glass transition temperature (Tg) which is the temperature at which the adhesive sample become soft. Previous researchers studied the thermal behavior of materials (Jinen, 1988; Bhowmik *et al.* 1998; Xie *et al.* 2005; Ratna *et al.* 2001). The mechanical performance of the adhesive is another important factor for evaluating the performance of an adhesive. Several works shows that increasing liquid modifier to epoxy adhesive might reduce the mechanical properties of the adhesive (Rinde *et al.* 1980; Sanjana & Testa, 1985; Huang & Kinloch, 1992; Jang & Yang, 2000; Paul & Bucknall, 2000; Pearson *et al.* 2000; Chikhi *et al.* 2002; Gam, 2003; Atefi *et al.* 2012a; 2012b; Davoodi *et al.* 2012) Therefore one should not add a large amount of liquid rubber to epoxy adhesive system. In this regards investigators should investigate all aspects of physical behavior and artifacts of epoxy-based modified adhesive.

In this work an epoxy adhesive is studied and the modification of adhesion is preformed using Hycar modifier. This is mixed with epoxy before mixing with hardener. We focus on thermal behavior of the adhesion using DSC. The glass transition temperature of the formulations is measured in this study. Moreover we evaluated the mechanical behavior of the adhesion using Impact test and Flexural test. In addition, scanning electron microscopy (SEM) is utilized in this work for better understanding the behavior of adhesive. The obtained data can be comprehensively useful for further manufacturer, investigators and industrial engineers who have investigated the physical performance of modified epoxy adhesives.

(a)



(b)

Figure 1. Use of adhesives in medical applications in (a) stopping bleeding and (b) surgical operation

2. Experimental

2.1 Materials

Two materials have been used: one is epoxy adhesive and the other is Hycar additive. Adhesive was made of two compounds: EPON828 epoxy resin and the Epikote DX6509 curing agent. These materials have been obtained from Shell Co. The adhesive is mixed in the ratio 80 part of EPON828 with 20 part of Epikote DX6509. The Hycar used is obtained from the Shimi-Mobtaker-Peivand (Islamic Republic of Iran) for modifying the adhesive material. This is a liquid state material that is added commonly to the adhesive for improving the adhesion properties of the epoxy adhesive

2.2 Sample preparation

First 0 to 24 percent of Hycar was appropriately weighted and nine samples are made (We present formulations that are used in Table 1). Then these amounts are added and mixed with EPON828. Mixing is done to obtain uniform composition. Afterwards, appropriate amount of Epikote DX6509 has been added to the mixture and we mixed it again for several minutes. Then mixture was cast into an appropriate small can. Afterwards, this is heated one hour at eighty centigrade degree. Specimens were cut out from the prepared by cutter.

2.3 Thermal property measurement

In this work the glass transition temperature is applied as an indicative for thermal strength of adhesives. A Differential scanning calorimetry (DSC) is used based on a Setaram DSC machine. Samples with weight of 8 mg are weighted and pressed in aluminum cans. Then these samples are heated from ambient temperature till 130 centigrade degree. Finally, these are cooled back to room temperature. The glass transition temperature (Tg) of the samples were measured.

2.4 Flexural Test

Flexural samples with a gauge length of 50 mm were made cutting from molded sheets and both sides were polished by sand paper until all visible defects disappeared. Flexural tests were performed at a strain rate of 0.5 mm/min at room temperature using an Instron universal machine. The extensometer was utilized to obtain accurate data.

2.5 Impact Test

Izod impact test was accomplished according to ASTM D256 using an 1 J hammer energy. Samples were prepared and machined to the standard shape ($62 \times 12.7 \times 4.2$ mm^3). Specimens were tested using at room temperature using an Impactor machine.

2.6 Microscopy

Fracture surfaces of the specimens were examined using scanning electron microscope (SEM). SEM samples were coated with a thin layer of gold before examination to protect the fracture surfaces from beam damage and also to prevent charge build up.

3. Results and discussion

It should be noted that different criterions are suggested by standard test methods for evaluating the thermal resistance of a glue or adhesive material. One important of these criteria is the Differential Scanning Calorimetry (DSC). DSC is used to measure glass transition temperature (Tg) which is the temperature at which the adhesive sample become soft. This property of a given plastic material is applied in many aspects of product design, engineering, and manufacture of products using thermoplastic components. Thermal simulations of a system will show temperatures that will be encountered by a specific component of that system. Knowing what temperature that a specific component will have to endure during use will allow the determination of the best material for that application (www.Wikipedia.com). The results of Tg measurement has been presented in Table 2. This table reveals that the glass transition temperature of adhesive decreases as the amount of Hycar increases. This is very significant for contents higher than 6%.

An interesting finding is that one can conclude to not increase the Hycor content beyond the critical value due to the possibility of diminishing the efficiency of the adhesive for high temperature applications. It is well-established that using modifier can improve the adhesion properties of glues (Bascom *et al.* 1975; Jinen, 1988). However, the modifier can reduce the thermal stability of adhesive.

Table 3 shows the flexural properties of epoxy as a function of Hycar content. As expected, the flexural strength radually decrease with increasing rubber content (Table 3). This is due to the fact that the strength and modulus of Hycar is much lower than that of the epoxy matrix. However note that the decreasing amount is not very considerable and therefore one conclude that the flexural strength remains constant over the wide range of Hycar.

The results of impact energy absorption (impact strength) versus Hycar content for notched test specimens are reported in Table 4. Note that modifier increases impact strength (Table 4). However for obtaining optimum physical properties, one should not use more than 9-12 % Hycar for modification of epoxy adhesive.

Table 1. Samples used

Sample Material	EP	EP-3	EP-6	EP-9	EP-12	EP-15	EP-18	EP-21	EP-24
EPON828	80	80	80	80	80	80	80	80	80
Epikote DX6509	20	20	20	20	20	20	20	20	20
Hycar	0	3	6	9	12	15	18	21	24

Table 2. Tg of formulations

Sample	EP	EP-3	EP-6	EP-9	EP-12	EP-15	EP-18	EP-21	EP-24
Tg	84.44	84.21	83.86	81.22	80.13	79.45	78.31	76.95	73.4

Table 3. Flexural strength

Sample	EP	EP-3	EP-6	EP-9	EP-12	EP-15	EP-18	EP-21	EP-24
Strength	105	104	105	103	105	102	103	102	101

Table 4. Impact strength

Sample	EP	EP-3	EP-6	EP-9	EP-12	EP-15	EP-18	EP-21	EP-24
Strength	2.4	2.8	3.3	3.8	4.1	3.6	3.1	3.2	2.9

Figure 2. SEM of neat epoxy without Hycar

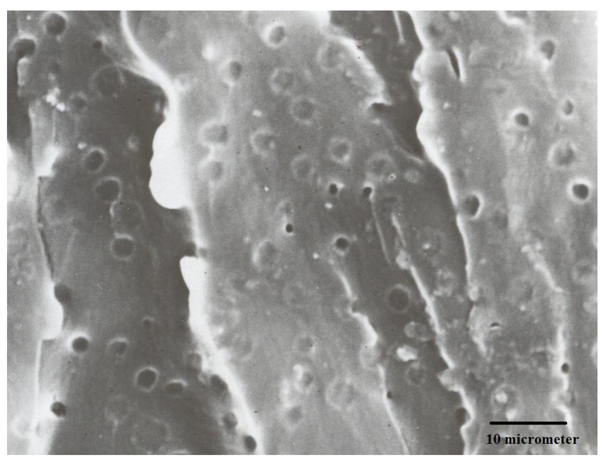


Figure 3. SEM of epoxy containing Hycar

Figures 2 and 3 shows the SEM fracture surface of specimens. Comparison between Figure 2 and Figure 3 reveals less roughness on the damaged surface of the Hycar modified epoxy specimen. Decrease in roughness corresponds to decrease in rigidity and increasing in energy absorption. It is in accordance with the Impact strength of Hycar-modified specimen in comparison with neat epoxy.

4. Conclusions:

Glues and adhesives are of important applications in medicine such as disposal medical devices, structural bonds, bone cement, prostheses, etc. In this paper the physical response i.e. thermal and mechanical behavior of the adhesive modified with liquid rubber has been investigated. In addition, scanning electron microscopy (SEM) is utilized in this work for better understanding the behavior of adhesive. Results show that adding liquid rubber decrease the Tg of the system. This does not change the flexural strength of the adhesive system. Using liquid rubber increase the Impact strength of the adhesive system. For obtaining an optimum physical properties, one should not use more than 9-12 % liquid rubber for modification of adhesive. The obtained data can be comprehensively useful for further investigators and medicine.

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