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## Effect of tackifier and crosslinkers on electron beam curable polyurethane pressure sensitive adhesive

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### ABSTRACT

Polyurethane based pressure sensitive adhesive using monofunctional urethane acrylate and difunctional urethane acrylate has been made using electron beam irradiation. The effect of varying electron beam doses on the adhesion properties viz., peel adhesion, shear adhesion and initial tack has been studied. Effect of tackifier and crosslinkers viz., polymeric methylene diphenyl diisocyanate, carbodiimide modified methylene diphenyl diisocyanate and triallyl cyanurate on adhesion properties as well as on moisture vapor transmission rate and gel content of the polyurethane based pressure sensitive adhesive has also been studied. It was found that with increasing dose, tackifier and crosslinkers concentration, all the adhesion properties viz., peel adhesion, shear adhesion strength and initial tack were increased upto a certain dose, concentration of tackifier and crosslinkers, reached a plateau and then levelled off with further increasing the dose, tackifier and crosslinkers concentration. Similarly, moisture vapor transmission rate was decreased and the gel content increased with increasing dose of electron beam.

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### 1. Introduction

Pressure sensitive adhesives (PSAs) are unique in nature and invariably used in almost every day life for labels, tapes, films and many more special applications (Yang-Bae, 2008; Kim et al., 2006; Satas, 1999; Bendek and Heymans, 1997; Varanese, 2000). PSAs are different from general adhesives as they can be attached or detached several times from the surfaces with a slight pressure and leaving no residue behind.

PSAs are made in different forms viz., solutions, emulsions and hot melts. Similarly, they have different mechanisms of curing. The predominantly used thermal curing method has many disadvantages, moreover, due to stringent environmental regulations, thermal curing which involves emission of volatile solvents in the environment has slowly been replaced, to a large extent by ultra violet (UV) curing (Allen, 1985). However, due to the associated limitations of UV curing, electron beam (e-beam) curing has emerged as a powerful tool for manufacturing of PSAs. One of the advantages of e-beam curable adhesives is that they do not use photoinitiators and thus, the possibility of degradation products of photoinitiators after curing is eliminated. This way,

the PSAs can be designed for the special applications particularly for the medical field. The moisture vapor transmission rate (MVTR) is an important requirement for adhesives used in medical applications. The MVTR helps to understand the transmission behavior of moisture vapor from the skin. A required level of MVTR not only helps in constantly pushing the moisture vapor out of the skin but also helps in retaining the adhesion strength for a longer time, because the accumulated sweat otherwise debonds the adhesive from the skin surface.

Polyurethane (PU) due to its excellent adhesion properties is widely used in industry, especially in the form of emulsions. The major share of these adhesives is in the packaging and lamination industry. Radiation curable polyurethane dispersions are also used in coating industry predominantly for wood, furniture and parquets (Tielemans and Roose, 2008). All of such PU dispersions are even though UV curable, curing by e-beam is considered more advantageous because of its efficiency and throughput.

PU has very good thermal, scratch resistance and physico-mechanical properties, which can be tailored for the required applications. PU is usually cured with chemical curing agents such as polyisocyanates at elevated temperature (Huang et al., 1992). However, chemical curing of PU has disadvantages, viz., the reaction is generally unpredictable and highly sensitive to temperature variations, moisture and stoichiometry. Moreover, the crosslink density and uniformity of the polymer is below

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desired levels. As a result e-beam curing has been attracting much attention as an alternative.

PSAs are characterized by their adhesion properties in terms of their ability to wet the surface and adhere quickly (tack), resistance to removal by peeling from an attached surface (adhesion) and ability to hold in position when shearing forces are exerted (cohesion). In the present work, acrylated polyurethane having ethylenically unsaturated group at the end i.e., monofunctional urethane acrylate (MFUA) as well as difunctional urethane acrylate (DFUA) were used and effect of tackifier and crosslinkers viz., polymeric methylene diphenyl diisocyanate (PMDI), carbodiimide modified methylene diphenyl diisocyanate (CMDI) and triallyl cyanurate (TAC) was studied on adhesion properties by irradiation with e-beam on doses ranging from 5–60 kGy.

## 2. Experimental

### 2.1. Materials

Genomer 4188, a monofunctional aliphatic urethane acrylate oligomer with 2-ethylhexyl acrylate and Genomer 4269 a difunctional aliphatic urethane polyester acrylate in combination with 2-[[butylamino]carbonyloxy]ethyl ester and 2-propenoic acid were procured from Rahn USA corp., USA. The MFUA has a  $T_g$  –16 °C, flash point > 83 °C, viscosity 26,000 cPs, density 1.05 g/ml, and water solubility < 1 g/l. The DFUA has a  $T_g$  –15 °C viscosity 21,000 cPs and density 1.1 g/ml and water solubility < 1 g/l. Both the acrylates were used as such as base resins without further purification in the adhesive formulations.

Genomer 6043, an inert modified saturated polyester resin with a combination of 2-[[butylamino]carbonyloxy]ethyl ester and 2-propenoic acid having  $T_g$  –18 °C, flash point > 100 °C, viscosity 19,000 cPs, density 1.13 g/ml and water solubility < 1 g/l was procured from Rahn USA corp., USA and used as such as a tackifier without further purification in the adhesive formulations.

PMDI having methylene diphenyl diisocyanate (MDI) 43%, NCO 32%, viscosity 50 cPs, M.Wt 340, density 1.234 g/cm<sup>3</sup>, functionality 2.7 was procured from Dow Chemical Company USA. TAC functionality 3 was procured from Sigma-Aldrich while CMDI having MDI 73%, NCO 29.2%, viscosity 33 cPs, functionality 2.1 was procured from Dow Chemical Company USA. All these crosslinkers were used as such without further purification in the formulations. Fumed silica (Cab-O-Sil) with particle size 5–50 nm, surface area 5–600 m<sup>2</sup>/g and density 2.2 g/cm<sup>3</sup> was procured from Cabot, USA and used as wetting agent in adhesive formulations.

### 2.2. Methods

PSA compositions were made using various combinations of tackifier and crosslinkers with MFUA and DFUA. In order to evaluate the PSA for its adhesion properties a PSA tape was made by depositing 0.2 mm thick and 30 g/m<sup>2</sup> adhesive coating layer on a release paper and left for air drying at room temperature for 30 min. The adhesive layer thus formed was ultimately transferred to the non-oven fabric to make the PSA tape. The thickness of non-woven fabric was 0.32 mm while its weight was 90 g/m<sup>2</sup>.

The dried tape samples were irradiated in air at Bhabha Atomic Research Center, Mumbai, India, by an e-beam accelerator ILLU-6. The accelerator has 2.0 MeV energy level with a conveyor speed of 3 cm/s for 10 kGy/pass and 6 cm/sec for 5 kGy/pass, with a pulse current of 300 mA, average current 2 mA and pulse frequency of 15 Hz. The samples were irradiated at different doses starting from 5 to 60 kGy. The irradiated samples were further exposed to dry air for 5–7 min before packaging.

The adhesion properties of dry as well as wet samples (by immersing in 1% saline solution for 8 h) were carried out for peel, shear adhesion and initial tack. The peel and shear adhesion studies were carried out using a Universal Testing Machine (UTM), Star Testing System, Mumbai. The samples for peel adhesion were prepared as per specification (ASTM D3330, (1993)) and conditioned at 25 °C and relative humidity (RH) 60% for 24 h before testing. The peel adhesion strength was carried out from a sample of size 300 mm × 20 mm with a glass plate at 180° angle. Contact area of the adhesive on the glass plate was 100 mm × 20 mm, gage length 150 mm and speed of the UTM was 500 mm/s.

The samples for shear adhesion for both dry and wet systems were prepared as per specification (ASTM D3654, 1988). The size of Samples was 200 mm × 20 mm with contact area of 50 mm × 20 mm on the glass plate, while gage length was 150 mm. The shear adhesion strength was carried out on a UTM with a speed of 200 mm/sec.

The initial tack studies of both dry and wet samples were carried out as per specification (ASTM D3121, 1994), which deals with the measurement of comparative tack of PSA tape by a rolling ball method. The size of samples was 400 mm × 50 mm with contact area of 380 mm × 50 mm, while the diameter of the steel ball was 10.96 mm and weight 5.47 g. The steel ball was rolled down with an inclination angle of 45° on the adhesive tape facing the adhesive side and the distance traveled by the ball was measured. The resistance force required to stop the steel ball on the adhesive tape was calculated in N/m.

Moisture vapor transmission rate of PU-PSA tape was determined as per specification (ASTM D3833M, 1988). A cup shaped aluminum disk with a diameter of 8 cm was filled with weighed quantity of dry CaCl<sub>2</sub> and the top of cup was covered with PSA tape and sealed from peripheries by hot paraffin wax. Initial weight of disk containing CaCl<sub>2</sub> and tape was taken before placing in the incubation chamber at a RH 70% and temperature 30 °C for 24 h. The MVTR was determined as follows:

$$\text{MVTR (g/hr-m}^2\text{)} = \frac{(W_2 - W_1) \times 24}{T \times A}$$

$W_1$  is the initial weight (g) of system;  $W_2$  the final weight (g) of system,  $T$  the time of exposure (h) between  $W_1$  and  $W_2$ ;  $A$  = area of cup opening (m<sup>2</sup>).

The gel content was determined by weight differential method. The irradiated polymer was dissolved in THF at 10% concentration in a closed flask and left at room temperature for 4 h. During this period crosslinked polymer swells while uncrosslinked portion is dissolved in solvent. The soluble portion of the polymer is recovered from the solvent by evaporation and gel content is determined by weight difference from the quantity of original polymer dissolved as following:

$$\text{Gel content(\%)} = 100 - \frac{W}{W_0} \times 100$$

where  $W$  is weight of polymer recovered after evaporation of solvent,  $W_0$  is initial weight of sample taken.

All the results were reported for an average of five samples for all of the above mentioned studies.

### 2.3. Chemistry of PU-PSA

The chemistry of PU-PSA involves preparation of a –NCO terminated prepolymer by reacting a molar excess of diisocyanate with a diol. The –NCO terminated PU-prepolymer is then reacted with an unsaturated hydroxy alkyl ester for making a urethane acrylate. Various crosslinkers were used for making the adhesive.

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