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Study of Polymer Binders to Develop Low Emissive Paints for Thermal Radiation Management Applications

Om Dev*, Shanker Dayal, Ashish Dubey & Alok K Dixit

Directorate of Stealth Materials and Technologies (DSMT), Defence Materials & Stores Research & Development Establishment, Kanpur- 208 013, India.

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The management of thermal emittance always possess challenges in both military and civil domains. The low emissive materials in form of paints offers viable solution for minimising thermal emittance of military objects for camouflage and enhancing infrared reflectivity of roof to reduce cooling loads. Binder apart from pigments is one of the critical components of low emissive paint formulation to decide its ultimate performance. For selection of appropriate resin and binder, a comparative study of five prevalent polymers *viz*. polyurethane (PU), poly vinyl chloride (PVC), polyethylene (PE), polypropylene (PP) and poly methyl methacrylate (PMMA) has been carried out. Polymeric film of thickness 100µm of each of the polymer is prepared by Elcometer 4340 motorized film applicator. Infrared spectra and thermogravimetric profile have been obtained by Bruker make Tensor 27 FTIR in spectral range 4000 – 600 cm^{-1} and TGA Q 500. Film formation of PU is excellent and transmission in 3-5 µm is almost 100%. Hence, it may be chosen as binder in low emissive paint for MWIR band. PVC and PMMA among the polymers offer excellent transmission 87-99% in both thermal transmission windows 3-5 µm and 8-12 µm. Thermogravimetric (TG) profile of polymer films evident of thermal stability of PVC, PMMA and PU polymers up to 200 °C. Infrared transmission, TG profile and film formability manifest that both binders- PVC and PMMA are suitable for development of low emissive paints applicable up to temperature 200 °C for both IR transmission windows to evade thermal detectors working for detection of military objects and reducing cooling load in buildings by enhancing reflectivity of roof by application of low emissive paint.

Keywords: Low emissive coatings; Thermal signature; Temperature control; Camouflage and stealth; Cool roof

1 Introduction

Solar radiation is essential for human race and spreads on entire earth. Around 53% of solar energy occurs in thermal infrared region, wavelength range 700- 2500 nm of electromagnetic spectrum[1-2]. This fraction of solar radiation increases temperature of exposed surfaces –residential buildings, commercial spaces, ships and military objects. The environmental conditions in summer having temperature range of 35-45 ºC are not comfortable for human and cause illnesses, epidemics and even death. A roof with high solar reflectivity (the ability to reflect sunlight) remains cool in the summer and reduces cooling demand. It reduces not only electric power consumption in air-conditioned buildings but also enhances comfort level in unconditioned buildings[3-5].

Camouflage and stealth in multispectral region of electromagnetic spectrum of military targets is indispensable for armed forces in present scenario. Detectors operating in thermal infrared region plays decisive and inevitable role in target Detection, Recognition and Identification (DRI) of military objects. The infrared (IR) detectors make use of two atmospheric transmission windows 3-5µm and 8-12 µm for thermal signature measurement for target detection and surveillance. Apparent temperature difference (ΔT) of target with respect to background plays key role in target detection[6-7]. Target detection probability decreases with reduction of ΔT.

Temperature reduction is necessary for decreasing detection probability of military objects[8] and heating load of solar radiation in buildings to cut down power consumption and enhancing comfort level. Low emissive paint/coating is a passive and cost effective solution for both requirements, which provides high reflectivity or low emissivity in thermal infrared region of solar spectrum[9]. Low emissive paint is composed of infrared reflecting pigments, resins/binders and additives. Infrared reflecting pigments and binders are key ingredients of low emissive paint formulation whose properties decide

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*Corresponding author:
(E-mail: omdev@dmsrde.drdo.in; omdevgangwar@gmail.com)

its ultimate performance and application. Resins and binders used in general purpose paints have high infrared absorption that makes them inappropriate for low emissive paints[10]. Optical properties in desired wave band, film formation, adhesion, binding of pigments and glass transition temperature are key properties of binders/ resins for paint applications[10-11]. Anderson *et al.* outlined properties of materials including pigments and binders for spectral design coatings in signature management applications[12]. Hallberg *et al.* & Wake *et al.* reported on the impact of binders in low emissive paint for camouflaging in 3-5µm and 8-12µm and suggested low absorption in thermal infrared band of resins and binders is essential for low emissive paint[10,13]. Infrared properties of various binders- polyethylene, vinyl polymers, epoxy, polyvinylidene fluoride, dimethyl silicone and water solvable butyl acrylate or methyl acrylate copolymers have been studied by several researchers for development of low emissivity paint and coatings[10,14-16]. Rosi *et al.* analysed infrared reflection properties of acrylic emulsion, polyvinyl acetate resin, oil-modified alkyd resin for their absorption bands and their potential overlaps in the mid and near infrared regions[17]. Most studies on IR properties of polymer binders for low emissivity paint were not analysed for thermal stability and temperature at which it to be apply on surface.

The present work is focused on comparative study of polymer binders for low emissive paints applicable at normal room temperature and their thermal stability. Five polymers- polyurethane (PU), poly vinyl chloride (PVC), polyethylene (PE), polypropylene (PP) and poly methyl methacrylate (PMMA) are taken for study. Film of each of the polymers has been prepared by electrically heated Elcometer 4340 motorized film applicator. Thermogravimetric behaviour of each film has been observed by TGA Q 500 (TA Instruments) and IR transmission measured in spectral range 4000 - 600 cm^{-1} (2.5-16.6 µm). The purpose of the study is to select appropriate binder for low emissive paint/ coating formulations for ground based military targets and cool roof applications.

2 Materials and Film Preparation

Paint is composed of pigment, resin/binder and additives. The pigment provides shade and optical properties, resin binds pigments and forms film on the substrate and additives impart additional properties[11]. Infrared reflecting (IRR) pigments and binders are most influencing ingredients for low emissive coating/ paint. The binder adds several properties to paints- adhesion, anti-scratch, durability, thermal stability and paint application temperature etc. The selection of appropriate binder is very crucial for end use.

2.1 Materials

Binders used in general have significant high IR absorption due to the presence of specific functional groups C-H, C-C and C-O[10,18]. Several sharp resonances occur at various wavelengths in long chain polymer molecules due to excitation of presented bonds by absorption of IR radiation. These polymers thus reduce the effect of IRR pigments and hence are not right choice for low emissive paints. Five typical polymers were taken in the present study, which are shown in the Fig. 1. Polyethylene (Low density, granular size 1000 µm, white colour, Alfa Aesar), polypropylene (granular size 3-4 mm, white colour, Alfa Aesar), poly vinyl chloride (granular size ~ 100 µm, white colour, Motley Exim) and polyurethane (granular size 3-4 mm, white colour, Lubrizol) polymers were in granule form and poly methyl methacrylate was in sheet form.

2.2 Film preparation

Paints involve the conversion process from liquid to solid form during application. The conversion process is the result of (a) drying of polymer solution (removal of solvent), (b) growth of finite molecular weight polymer to an infinite polymer network through chemical reaction, or combination of (a) and (b). Large polymer chain can form entanglements with other chains and they can condense with

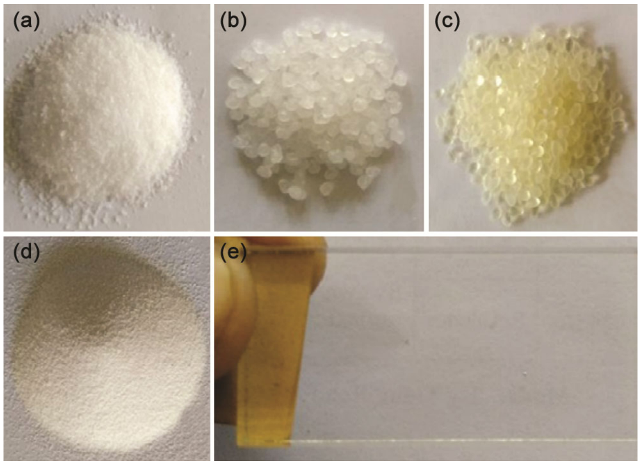

Fig. 1 — Polymer resins (a) PE, (b) PP, (c) PU, (d) PVC and (e) PMMA.

themselves and their neighbours under the pressure of vanderwaal forces. In some cases, reactive groups may form chemical bonds between adjacent chains. These are examples of crosslinking by which chains attach among each other, so that force may be transferred from one chain to another. The adequate crosslinking forms an infinite network makes mechanically enough capable against applied stress.

The polymer binders are widely used as coating materials for textile, leather, construction materials and metallic surfaces[19]. For thermal camouflage and stealth applications, resin with fillers of desired properties in form of paints and coatings in different camouflage shades (dark green, olive green, dark brown and sand *etc*.) is used[11,20]. These paint formulations form film on fabric and surface of military objects that reduces thermal emittance and enhance solar reflectance of roof of buildings. Polymer solution has been prepared using IKA make magnetic stirrer at 900 - 1400 rpm. The chemical details and set temperature of stirrer for each binder are given in Table 1. The film of each polymer of thickness 100 μm was prepared by electrically heated Elcometer 4340 motorized film applicator. During film preparation, traverse speed of doctor blade was 5mm/sec. The polyethylene, polypropylene solutions were poured on coating bed at 100 ºC and its temperature reduced by 10 ºC in 5 minutes time interval till room temperature attained. Polyurethane, poly vinyl chloride and poly methyl methacrylate films were prepared at room temperature. The circular portion of each film of size 40 mm diameter is cut out by sample cutter. These films are shown in the Fig. 2

3 Interaction of radiation with polymers

Infrared radiation is a part of electromagnetic spectrum situated between visible and microwave region. The spectral distribution of radiation of a perfect thermal radiator is governed by Planck's law

$$W_\lambda = \frac{2\pi hc^2}{\lambda^5}\frac{1}{e^{hc/\lambda kT}-1} \quad \dots (1)$$

Table 1 — Binder, solvent and temperature.

Binder	Solvent	Polymer (gm) / solvent (mL)	Temp for solution preparation (ºC)
Polyethylene (PE)	MEK	10/40	80- 100
Polypropylene (PP)	Xylene	10/40	80- 100
Polyurethane (PU)	MEK	10/40	35 - 39
Poly Vinyl Chloride (PVC)	THF	10/40	35 - 39
Poly Methyl Methacrylate (PMMA)	Acetone	10/40	35 - 39

Fundamental EM theory states that intensity of EM radiation attenuates exponentially during its propagation through polymers (optical materials) due to complex refractive index (n-jk). Here, n and k are real and imaginary part respectively of refractive index of media. This exponential attenuation caused by absorption in material is defined as

$$\alpha\,(\lambda) = 4\pi k/\lambda \quad \dots (2)$$

Where λ is wavelength of radiation.

An infrared active molecule undergoes dipole moment change during vibration. The prime vibration mode of infrared active molecules are stretching and bending. The interaction of IR radiation with film is shown in Fig. 3.

When IR radiation of intensity $I_o(\lambda,\theta)$ incidents obliquely on the polymer film of thickness 'd', then reflection and transmission occur simultaneously at air-polymer interface. At this interface component $rI_o(\lambda,\theta)$ reflects back and remaining part $(1-r)I_o(\lambda,\theta)$ enters into film. The entered beam is attenuated exponentially within film. This attenuation depends on optical properties of materials and obeys Beer's law. If absorption coefficient and path length in the material are $\alpha\,(\lambda)$ and x respectively, then intensity of

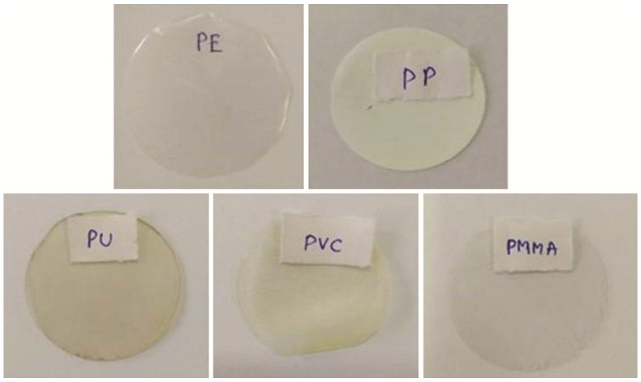

Fig. 2 — Polymer films of- PE, PP, PU, PVC and PMMA.

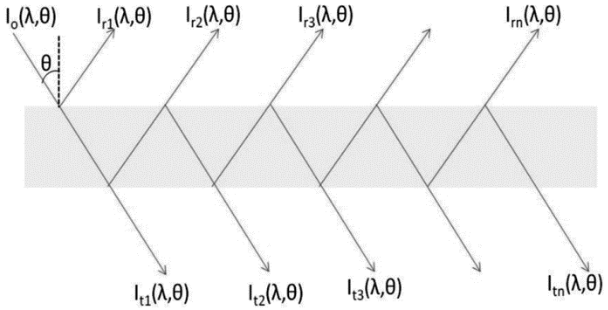

Fig. 3 — Interaction of IR radiation with film.

incident radiation just before crossing the second polymer-air interface will be $(1\text{-}r)I_o(\lambda,\theta)e^{-\alpha(\lambda).x}$. This attenuated beam at second polymer-air interface undergoes further reflection and transmission. At this instant, the emerging intensity will be $(1\text{-}r)^2I_o(\lambda,\theta)e^{-\alpha(\lambda).x}$. Reflected intensity $r(1\text{-}r)I_o(\lambda,\theta)e^{-\alpha(\lambda).x}$ from the second interface will undergo further multiple reflection, transmission and absorption as shown in Fig. 3.In this process of Fresnel reflection, the incident and refraction angles are assumed to satisfy Snell's law. If reflection coefficient of air-polymer film interface is r, then total reflected $I_r(\lambda,\theta)$and transmitted intensity $I_t(\lambda,\theta)$ will be given by

$$\mathrm{I_r}(\lambda,\theta) = \mathrm{I_{r1}}(\lambda,\theta) + \mathrm{I_{r2}}(\lambda,\theta) + \mathrm{I_{r3}}(\lambda,\theta) + \quad \ldots(3)$$

&

$$\mathrm{I_t}(\lambda,\theta) = \mathrm{I_{t1}}(\lambda,\theta) + \mathrm{I_{t2}}(\lambda,\theta) + \mathrm{I_{t3}}(\lambda,\theta) + \quad \ldots(4)$$

Where $I_{r1}(\lambda,\theta)$,$I_{r2}(\lambda,\theta)$…..and $I_{t1}(\lambda,\theta)$, $I_{t2}(\lambda,\theta)$….. are components of reflected and transmitted intensities from first and second interface respectively. Hence, reflectance of film will be

$$\mathrm{I_r}(\lambda,\theta) = \mathrm{rI_0}(\lambda,\theta)\left[1 + \frac{(1-\mathrm{r})^2\,\mathrm{e}^{-2\alpha(\lambda)\mathrm{d}\,\sec\theta_\mathrm{r}}}{1-\mathrm{r}^2\mathrm{e}^{-2\alpha(\lambda)\mathrm{d}\,\sec\theta_\mathrm{r}}}\right] \quad \ldots(5)$$

Now, the transmittance$\mathrm{I}_t(\lambda,\theta)$ of film will be

$$\mathrm{I}_t(\lambda,\theta) = (1-r)^2\mathrm{I_0}(\lambda,\theta)\frac{\mathrm{e}^{-\alpha(\lambda)\mathrm{t}\sec\theta_\mathrm{r}}}{1-\mathrm{r}^2\mathrm{e}^{-2\alpha(\lambda)\mathrm{t}\sec\theta_\mathrm{r}}} \quad \ldots(6)$$

In equations 5-6, refraction angle θ_r is

$$\theta_r{=}\sin^{-1}\left(\frac{\sin\theta}{n}\right) \quad \ldots(7)$$

For normal incidence (θ=0°), reflection loss coefficient for air-polymer interface is given by $r = [(n-1)^2 + k^2]/[(n+1)^2 + k^2]$

For polymeric films, imaginary part is negligible compare to real part of refractive index. Hence, reflection coefficient of the interface will be

$$r = [(n-1)/(n+1)]^2 \quad \ldots(8)$$

Substituting value of refection coefficient in equations 5 and 6, spectral reflectance and transmittance can be obtained for normal incidence.

4 Measurements

4.1 Infrared transmittance

Spectral transmittance measurement of polymeric films of PE, PP, PU, PVC and PMMA has been performed by Bruker Tensor 27 FTIR (Fourier transform infrared) spectrometer in infrared region 4000-600 $\mathrm{cm^{-1}}$ (wavelength range 2.5-16.6 μm). When IR radiation propagates through the polymer film, then film absorbs radiation quanta of characteristic wavelength. The absorption spectrum is mapped in the form of changes in the IR spectra radiation intensity as a function of radiation energy for knowing spectral properties of materials. The spectral intensity of a beam after passing through film $\mathrm{I_t}(\lambda,\theta)$ and reflected back $\mathrm{I_r}(\lambda,\theta)$ is governed by equations 5 and 6. The spectral transmission behaviour of polymer film in thermal infrared region is most important attribute for development of low emissive formulations.

4.2 Thermogravimetric analysis

Thermal stability is an important property of paint and coating that is studied by thermogravimetric analysis (TGA). It measures amount and rate of change in the mass of a material as a function of temperature or time in controlled atmosphere. TGA Q 500 (TA Instruments) has carried out TGA of all polymeric films under nitrogen purge and heating rate 20 °C/min.

5 Results and discussion

FTIR spectrum of materials is based on the absorption of infrared radiation at frequencies matches with normal vibration modes of macro molecule. These absorption features are characteristic of the molecular configuration, sequencing, conformation, and state of order. The intensity of an absorption band is related to the dipole moment change associated with the molecular vibration[21]. The Fourier transform infrared spectra have been recorded with spectral resolution 2 $\mathrm{cm^{-1}}$. The spectral transmittance of low density PE, PP, PU, PVC and PMMA films are shown in Fig. 4-8.

Figure 4 shows IR transmittance of low-density polyethylene (LDPE) film having molecular structure unit $(\mathrm{CH2\text{-}CH2})_n$. The two absorption bands are observed at 3.43 and 3.51 μm in MWIR (3-5μm) and none absorption band is occurred in LWIR (8-12μm).

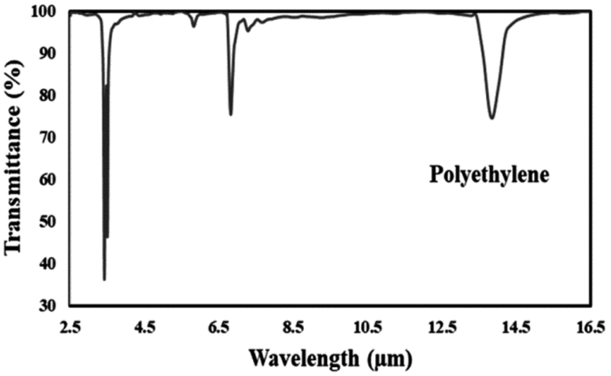

Fig. 4 — FTIR spectrum of low-density polyethylene.

These absorption peaks are result of asymmetrical and symmetrical stretching vibrational changes in (C-H) methylene group[22]. It is clear from Fig. 4 that the LDPE is almost fully transparent in 3-5µm and 8-12 µm IR transmission windows except two bands in 3-5µm. However, at room temperature, film formation is not possible due to ineffectiveness of molecular crosslinking and glassy state of solution.

The FTIR spectrum of polypropylene (PP) film, $(C_3H_6)_n$, is shown in Fig. 5. Each unit of PP molecule chain has three atoms of carbon in the form of different groups[18,23] -CH, CH_2 and CH_3. Each of them is related in IR spectra with characteristic absorption peak to definite wavelength. The strong absorption peaks observed in middle wave infrared (MWIR) region are 3.39, 3.43, 3.48 and 3.53µm while in long wave infrared (LWIR) region are 8.59, 10.02, 10.28 and 11.89 µm. The peaks observed in MWIR band are due to stretching vibrational changes of CH_3 and CH_2, first two peaks are due to asymmetrical stretching vibration while other belongs to symmetrical stretching vibration. The peaks observed in LWIR (8-12µm) band are 8.59- wagging vibration of CH_3 and 10.02, 10.28, 11.89µm- rocking vibration of CH_3, CH_2 & CH. The PP film offers excellent transmission ~97-98% in both 3-5µm and 8-12µm but film formation at normal room temperature is not possible due to ineffectiveness of molecular crosslinking and glassy solution.

Polyurethane is one of the important organic polymers for intensive technological applications. Fig. 6 shows the Fourier transform infrared spectrum of polyurethane film. The absorption bands at 2.99, 3.39 µm lies in MWIR while absorption bands at 8.21, 8.57, 9.38 and 10.48 µm are associated to LWIR. Different groups namely CH, CH_2, N-H, CN, C=O and C-O-C are associated with different absorption peaks observed in its IR spectrum. Fig. 6 depicts that polyurethane is almost 100% transparent in 3-5 µm except two weak absorption bands at 2.99 and 3.38 µm which are related to stretching of NH and CH_2 respectively while in 8-12 µm thermal region, it shows transparency approximate 70%. In this region, strong peaks observed at 8.21 and 8.57 µm are associated with CN group and other weak peaks are due to C-O-C group. During film formation, it was observed that polyurethane solution at room temperature shows excellent molecular crosslinking.

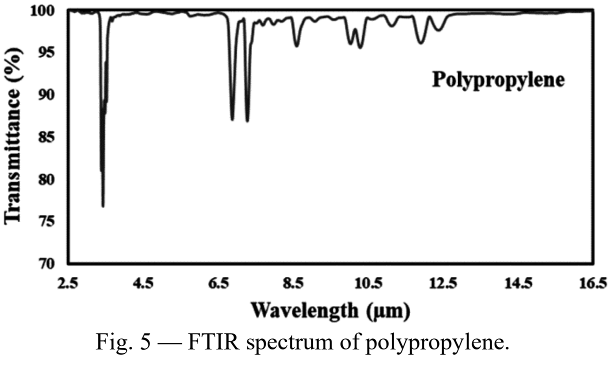

Fig. 5 — FTIR spectrum of polypropylene.

PVC polymer is extensively used in coated technical textile. FTIR spectrum of PVC is given in Fig. 7. Different vibrational groups- CH, CH_2, CO and C-Cl are associated with its characteristic peaks of IR spectrum[21]. In 3-5µm waveband, only one absorption band at 3.43 µm is present while in 8-12 µm observed strong bands are at 8.01 and 10.44 µm. The absorption band occurred in MWIR is due to stretching vibration of C-H. In LWIR region, observed strong bands at 8.01 and 10.44 µm are attributes of vibrational changes due rocking and wagging of C-H respectively. Fig. 7 shows that its transparency is 99 % in 3-5 µm and around 94% in 8-12 µm. PVC solution at room temperature shows excellent molecular crosslinking property and its film can be prepared at room temperature with excellent properties. The IR transparency and film formation ability is good enough for its usage as a binder for low emissive paints.

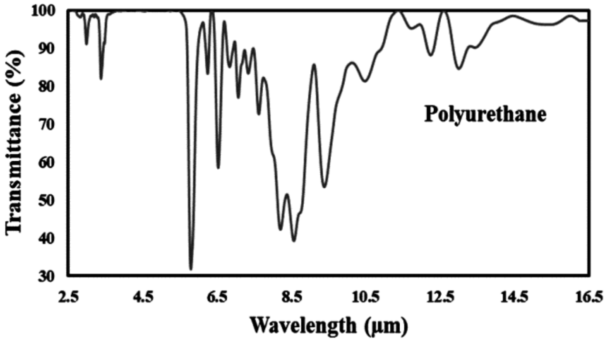

Fig. 6 — FTIR spectrum of Polyurethane.

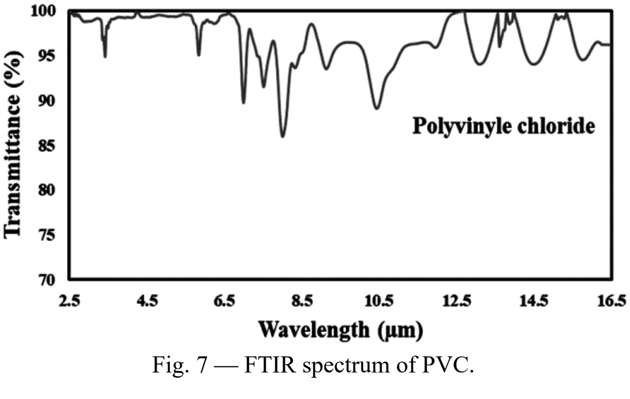

Fig. 7 — FTIR spectrum of PVC.

Poly methyl methacrylate (PMMA) has excellent mechanical properties. IR active groups of PMMA are O-CH_3, C-O and C=O[14,21]. Its IR transmission behaviour is shown in Fig. 8. The film is almost 100 % transparent in 3-5 µm except one peak appeared at band 3.39 µm which is resultant of change in dipole moment due to CH_2 stretching. In 8-12 µm thermal region, peak at 8.42 µm is assigned to –O-CH_3 stretching vibrations and broad peak 8.07-11.86 µm is associated with C-O and methyl group (ester bond) stretching vibration. Its transparency in 8-12 µm is around 87 %. Film formation capability is good enough at room temperature due adequate molecular crosslinking. Its IR transparency and film formability are attractive for low emissive paints.

Thermogravimetric profiles of PE, PP, PU, PVC and PMMA are shown in Fig. 9. The figure shows all the polymers start breaking at different temperature. No mass reduction is observed up to 150 ºC for all the samples which is sufficient temperature for ground based military targets and buildings. PVC and PMMA show their thermal stability up to 150 ºC. PU, PP and PE are showing higher thermal stability compare to PVC and PMMA.

Fig. 4-8 shows that all the polymers offer excellent transmission in MWIR than LWIR of thermal infrared region. It is due to presence of IR active groups and their response against radiation lies in 8-12 µm. Polyethylene and Polypropylene have offered excellent IR transmission in both IR windows and thermal stability but film formability at room temperature is not possible due high glass transition temperature and inadequate crosslinking. Therefore, both polymers are inappropriate for low emissive paint. Polyurethane polymer has thermal stability up to 300 °C and outstanding film formability at room temperature due to prominent molecular crosslinking. It offers 83-98 % transmission in MWIR and 70 % in LWIR. IR transmission, thermal stability and film formability of PU restrict it for 3-5µm only.

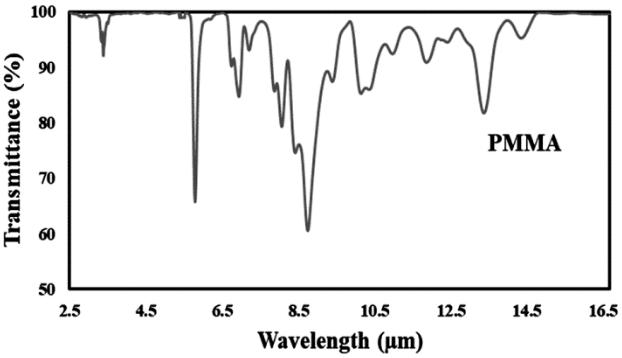

Fig. 8 — FTIR spectrum of PMMA.

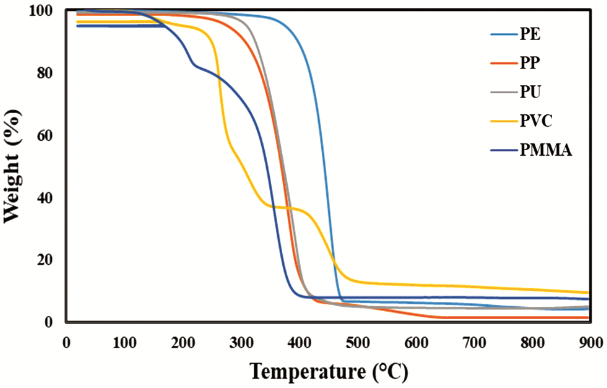

Fig. 9 — Thermogravimetric profiles of polymers.

Both PVC and PMMA offer excellent transmission 87- 99 % in 3-5 and 8-12 µm in which most IR surveillance/ detecting devices are working as well as enhancing infrared reflectivity of roof to reduce cooling load. Both polymers have thermal stability up to 150ºC and molecular crosslinking and film formability at room temperature of both PVC and PMMA are excellent. IR transmission of PVC is highest among the five polymers taken in present study. Result manifests that PVC and PMMA are appropriate polymer binders for development of low emissive paint in both 3- 5 µm and 8-12 µm aimed to control thermal emittance of objects up to temperature 150 °C. PU is also promising binder for 3- 5 µm band for emissivity paint aimed for higher temperature up to 300 °C.

6 Conclusion

The polymers- polyurethane (PU), poly vinyl chloride (PVC), polyethylene (PE), polypropylene (PP) and poly methyl methacrylate (PMMA), have been studied to explore binders for development of low emissive paints for thermal camouflage of military objects by evading infrared detection systems working in 3-5 and 8–12 µm transmission windows and to reduce cooling load of roofs by enhancing infrared reflectivity. Thermogravimetric profile, IR transmission in 3-5 µm and 8-12 µm of polymers and film formability at room temperature reveal that in spite of outstanding IR transmission, not all the polymers are suitable as binder for low emissive paints due to poor molecular crosslinking at room temperature. PP and PE are almost fully transparent in thermal region and thermally stable up to 300 °C or higher temperature but film formation at room temperature is not possible; hence these are not suitable as binder in low emissive paint. Film formability of PU is excellent and thermally stable up to 300 °C but average transmission in 3-5 µm is 90 % but it is poor 70 % in 8-12 µm; hence, PU may be useful as binder low emissive paint only for MWIR band. Both PVC and PMMA demonstrated excellent molecular crosslinking during film preparation and thermal stability up to 150 °C. PVC and PMMA offer 94-99 and 87-99 % transmission in 3-5 and 8-12 µm respectively. Both PVC and PMMA are suitable as binder for low emissive paint for thermal camouflaging of ground based objects and cool roof of buildings. PVC is most appropriate where coating hardness is not much significant parameter due to highest IR transmission among the five polymers.

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