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Research Article

THERMALLY STIMULATED DEPOLARIZATION CURRENT IN PURE AND SENSITIZED (WITH MALACHITE GREEN) POLYVINYL CHLORIDE

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ABSTRACT

The thermally stimulated depolarization currents (TSDC) in solution grown pure polyvinyl chloride and sensitized (with malachite green) PVC samples were studied as a function of temperature of polarization polarizing fields and the amount of malachite green. The TSDC spectra of pure PVC thermo electrets are characterized with two relaxation peaks located around 55 and 140°C respectively. As the amount of malachite green is increased the higher temperature peak shifts from its original position and appears at similar temperature in opposite polarity. The peak of opposite polarity is explained in terms of a schottky barrier field at the positively biased metal-dielectric contact interface and hetero/homo-charges. Increase in current on sensitization has been attributed to the formation of charge transfer complex due to the interaction between the sensitizer and the polymer matrix.

Key Words: *Thermally Stimulated Discharge Current (TSDC), Polyvinyl Chloride (PVC), Malachite Green, Schottky Barrier, Hetero and Homocharges, UV-VIS*

INTRODUCTION

After the universal acceptance of the two charge theory of Gross (Gross and Denard, 1948) for electrets, various attempts have been made to investigate the internal behaviour of the electret material responsible for charge storage and charge decay processes. Thermally Stimulated Discharge Current (TSDC) measurement is a novel technique to evaluate and identify the various processes operative during the storage and charge decay in the electret. The method involves the depolarization of the electret in absence of an external field while its electrodes are short circuited through a sensitive current detector. The current which appears in the external circuit due to depolarization processes is recorded as a function of time or temperature. Each depolarization processes, becomes detectable when its relaxation time is short enough by producing a depolarization current which increases at first with temperature, reaches a maximum and then drops to zero as the equilibrium distribution is achieved. TSDC has proved to be a loyal tool to investigate the molecular relaxation and charge storage behaviour of insulators including polymers. TSDC reveals the nature of various decay processes very quickly and precisely which confirms it as an attractive tool for determining the electrets behavior (Khare *et al.*, 1995). Other features responsible for the popularity of this technique are the convenience, high sensitivity and high resolving power.

Early experiments of TSDC were carried out without programmed heating with the main purpose to measure the total released charge (Frei and Groetzinger, 1936). However, since mid sixties, investigators have been using linear temperature rise and recorded current versus temperature values to obtain TSDC curves. Since then TSDC techniques have made tremendous progress in several laboratories and have been applied to the study of many dielectrics, ionic and divalent crystals, semiconductors, polymers, etc. Bucci *et al.*, (1966) studied TSDC in ionic crystals and formulated the first theory of TSDC for dipoles with single relaxation time. The theory has later been shown valid for charge migration over microscopic distances with subsequent trapping. It has, further been generalized by Gross and Electro (1968), Van Turnhout (1975) for the analysis of polarization with distribution in natural frequencies. Various polymers have been investigated for their charge storage properties. The shape of TSDC thermograms depends on the phenomenon causing the storage of charge and the area under the thermogram is proportional to the total charge stored in the electrets. However, apart from charge estimation, TSDC thermogram can also be used for obtaining information about the distribution function for relaxation time and activation energies, and also dielectric constant at ultra low frequencies.

The shapes of TSDC curves are sensitive to activation energy. Trap depth and relaxation times like internal parameters (Van and Ong, 1975) and heating rate, forming and storage temperature, forming time and field like experimental parameters. Valuable information regarding these internal parameters can, therefore, be obtained by a set of TSDC experiments with suitable experimental condition. Thus the advances made in TSDC studies have offered immense possibilities for studying various relaxation processes in pure and doped dielectrics (Gupta *et al*., 1979; Ikeda and Matsuda, 1979; Samoc and Samoc, 1979; Pillai and Rashmi, 1979). In general TSDC peak is always sharp for non distributed polarization. However, broadness of the peak is attributed to distributions either in relaxation time, or activation energy (Van, 1979; Vanderschueren and linkens, 1978). As such, it cannot be decided at first sight that the broad peak originated is due to distribution in polarization, or in activation energy, or both, however, it can easily be investigated by monitoring the peak position and slope changes in a set of TSDC thermograms pertaining to various experimental conditions.

The study of relaxation behaviour using TSDC of pure and sensitized (with malachite green) PVC is of considerable importance both from the technical aswell as from the fundamental point of view also the introduction of a specific impurity might assist in understanding the relaxation processes more clearly. The work reported in this paper elucidate that the dielectric relaxation in PVC not only depends on the structure but also on the presence of impurity. The effect of sensitizer on the pure polymer matrix was studied using TSDC.

The depolarization kinetic data i.e., activation energy, charge released and relaxation times for pure and sensitized PVC samples poled at fixed temperatures with various fields were calculated and listed in table.

MATERIALS AND METHODS

The polyvinyl chloride (PVC) and malachite green used in the present investigation were obtained from Glaxo Laboratory, Mumbai. The isothermal immersion technique was utilized for preparing pure and sensitized samples. The solution was prepared in a glass beaker by dissolving PVC and malachite Green in chemically pure dimethyl formamide (DMF). The solution was kept for 24h to get a homogeneous solution. The solution thus prepared was then poured onto an optically plane cleaned glass plate floating on mercury. The solvent was allowed to evaporate in an oven at 50ºC for 24h to yield the desired samples. This was followed by room temperature out gassing at 10–5 torr for a further period of 12 h to remove any residual solvent. The plate was then drawn slowly out of the solution, leaving a uniform film on the plate. Great care was taken to avoid air bubbles during the setting of polymer solution on the glass plates. The films were then detached carefully form the glass plate using a sharp edge knife of blade. Samples thus obtained were uniformly smooth and could be easily peeled from the glass surface.

The preconditioned samples were sandwiched between aluminized electrodes over a central circular area of 36 mm diameter. The thickness of the sample was of the order of 40 µm which was estimated by measuring the capacitance of the fabricated sandwiches.

The prepared samples were then sandwiched between aluminium electrodes inside an oven and heated to predetermined polarization temperature Tp. After achieving thermal equilibrium, a DC field i.e., polarization field Ep.was applied for 90 min. The sample was then cooled to room temperature under the applied DC field. Then the field was removed and sample was short circuited to remove stray charges. Now, the thermoelectret obtained was reheated at the rate of 3^{o}C per minute and the depolarization current was then recorded. The polarization was carried out by connecting a DC power supply (EC-HV 4800D) in series with the sample and with a Keithley 610C electrometer, which was carefully shielded and earthed to avoid earthing loops and extraneous electric noise.

To characterize the mechanism responsible for the spectra of pure and sensitized samples, effect of electric field stress Ep on the samples was observed. Two sets of samples were used for polarization and thermally stimulated discharge current (TSDC) studies. The first one termed A was pure polyvinyl chloride (PVC) and second set of those with malachite green of various concentrations was designated as P1, P2,P3 and P4 for 06, 7.5, 10 and 15% respectively.

The absorption spectra were record by UV-VIS spectrophotometer (PERKIN ELMER UV/VIS Spectrometer Mambda 12) in the wavelength range from 200 to800 nm. All the observations were carried out with the accuracy of ± 5% experimental error and results are reproducible.

RESULTS AND DISCUSSION

Thermally stimulated depolarization current (TSDC) spectra for A and P1, P2, P3 and P4 samples polarized with poling fields at 20, 30, 40 and 50 kV/cm at Tp = 90^{o}C are shown in Figures 1-4. The representative results for samples charged at 90^{o}C with various fields are reported. The absorption spectra for pure and sensitized PVC samples are shown in Figure 6.

The TSDC spectra of pure PVC samples consisted of two peaks, located around 55 and 140^{o}C respectively (Figure 1). However, on sensitization, the first peak shifts towards higher temperature and second peak shifts towards lower temperature and current starts appearing in the negative direction (Figure 2). For the higher concentrations both the peaks merge into one and the corresponding thermograms are characterized by peaks opposite in polarity observed around 65 and 140 ^{o}C (Figures 2, 3 and 4).

The UV absorption spectra of PVC sample is linear, however, sensitized PVC of different composition (Figure 6) show the existence of two sharp absorption bands at about 440 and 630 nm. The intensity of bands increases with respect to sensitization.

PVC is known to exhibit three relaxation processes, β, α and α`(Adamec and Kolloid, 1970). The β peak occurs in the glassy state of the polymer usually below room temperature and is due to the local reorientation of the polar side groups while the α peak is found at the glass rubber transition temperature Tg, where either the segment motion of the main chain, or the disorientation of the polar side group carrying a net dipole-dipole moment sets in.

The third process, α`, which is observed at temperatures higher than those for α process, is probable due to the charge accumulation (space charge) near the electrodes. In the present investigation, we have observed two peaks. The first peak observed just near the glass transition temperature of PVC corresponds to α relaxation process and the activation energy associated with this discharge process is 0.58 eV. The observed activation energy is comparable to that associated with the molecular motion or the phase transition process in PVC (Saito *et al.,* 1968), it must correspond to a molecular motion or dipole disorientation process rather than a trap depth further the observed peak must be related to the depolarization of the aligned dipoles connected with the chain of the polymer rather than to the charge released during the glass transition temperature. Because the magnitude of the charge corresponding to this peak increases linearly with an increase of Ep, a dipolar mechanism is involved during the depolarization process of peak first. The activation energy of 1.22 eV for the higher temperature peak (α`) is large and associated with an ionic mechanism rather that with a dipolar mechanism or some dipolar mechanism of some dipolar rotation. For this peak released charge show a saturation trend with increase in Ep.

The mechanism of ionic conduction in PVC, found by several workers (Kosaki *et al.,* 1971; Green, 1965; Seanor, 1967; Verma and Bhatnagar, 1975) and they confirm the existence of excessive ions in the bulk of the polymer. These ions must have come either from the dissociation of some of the molecules or from the thermal degradation of PVC (Rasuvaev *et al.,* 1971). Further confirmations obtained from the values of activation energy shown in Table-1 which are usually associated with ionic traps in amorphous materials (Chen, 1969; Jonscher, 1967). Initially these ions are distributed uniformly throughout the material. During the polarization the ions undergo a microscopic displacement and are trapped into the available traps. When the polymer is heated to observe the TSDC these ions are released from the traps, they migrate under the local fields and are recaptured.

At a particular temperature of polarization a certain definite number of carriers are available which, while moving under the action of the minimum applied polarizing field, may get trapped. Since this polarization is due to the migration of ions, it is a time-dependent phenomenon.

Because the charge carriers already present and the injected charge carriers both in a polymer are ionic in nature (Pfestorf, 1938; Mahendru *et al.,* 1978), their activation energy will be ≥ 1 eV and they can contribute only to the peak second or third(because the activation energy for the peak first has been evaluated to be about 0.58). Also no depolarization current was observed on heating the polarized specimen beyond160 ^{o}C, which indicates that the charge carriers either already present or injected (Gupta *et al.*, 1979) during depolarization into the material, or depolarized at temperature less than 160 ^{o}C. Therefore, it is assumed that α` peak is associated with the charge which is resultant of the charge carriers already present in the material and the injected charge carriers. Saturation trend of charge released verses Ep confirms above said statement (table 1).

The TSDC spectra are characterized by various peaks, due to dissipation of charges arising from polarization during charging process. The polarization of the material may arise due to various mechanisms namely, dipole orientation, charge displacement, molecular and domain structures, macro/microscopic displacement of ions with subsequent trapping, surface and space charge polarization etc. The processes other than the last two, give rise to the TSD current of polarity opposite to charging current (Ic). But in the case of surface and space charge polarizations current of opposite or the same polarity as that of Ic may be found depending upon the distribution of the space charge in the bulk of the sample. If the zero field point in the case of space and surface charge polarization is nearer to the electrode than the centre of the bulk, the current is supposed to be of the polarity opposite to Ic, same polarity of the Ic (Khare and Srivastava, 1994).

The appearance of a peak with polarity opposite to that of the dipoles suggests that, in addition to the normal mechanism of polarization described earlier, some other mechanism must be operative which traps the mobile charge carriers responsible for the negative polarity of the thermally stimulated discharge current. This necessitates some form of potential barrier which will have a built-in electric field opposite to that due to the applied polarizing voltage the Schottky (Jain *et al.,* 1978). Although such potential barriers could exist in the bulk of the material owing to in homogeneities of segregation of impurities, the Schottky barrier at the metal-dielectric contacts is more likely to be responsible for the 160 ^{o}C peak, which always has a polarity opposite to that of the normal TSC.

Polyvinyl chloride being a semi crystalline polymer, amorphous regions can act as traps, and these regions have energy levels different from those of the crystalline regions. The plasticizer and fillers etc used in the preparation of PVC may give rise to these energy levels as electron trapping is operative in both pure and doped samples of PVC. Also these plasticizers act as internal lubricant and supply charge carriers. Moreover, in PVC, electron traps are found to be neutral when empty and charged when filled (Talwar and Behari, 1981).

According to the two charge theory advanced by Gross (Gross, 1949), net apparent charge density of an electret is the combined effect of two charges, homocharge and heterocharge. The heterocharge is the volume charge produce by either the rotation of dipoles or the separation of the ions. The homocharge, on the other hand, is the space charge consisting of ions or electrons and is considered to be produced by the discharge in the minute air gaps between the electrode and the surface of the sample. An imperfect contact between the poling electrode and the surface of the sample increases the possibility of discharge in minute gaps and thereby producing the homocharge. The homocharge is also produced by the injection of charges from the poling electrode into the sample.

Neutralization of both homo- and hetero-charge carriers produces a depolarization current in the external circuit, but the directions of the currents are opposite. Due to the semi crystalline character of PVC a Maxwell-Wagner (MW) effect may build up at the interface of the textural elements in the polymer, giving rise to heterocharge. Polarized regions then attract the charge carrier of opposite polarity from either the bulk of the material, or those sprayed from the dielectric-electrode breakdown. These ions get settled on the surface of the molten dielectric giving rise to homocharge (Talwar and Behari, 1981). It appears that the peak in the negative direction is associated with the injection of charge carriers from the electrode in the presence of higher charging field. Addition of sensitizers increases the conductivity of polymer considerably at the poling temperature. High conductivity or reduced insulation capacity of sensitized samples may facilitate the injection of charge carriers directly from the electrode. The neutralization of these injected charge carriers caused by the increased conductivity at higher temperature will result in a peak in the same direction as charging current. Peak in the positive direction may also be attributed to the fast detrapping rate of charge carriers, exceeding the charge exchange rate of the injected electrode, thus resulting in partial blocking of the electrode during depolarization, resulting in shift of the position of zero field planes towards non-injecting electrode as suggested by Gross and Perlman (Gross and Perlman, 1972). Two charge reversals were also observed by Wiseman and Lindon (Wiseman and Linden, 1963) in case of polyvinyl acetate thermoelectret. According to them, the observed initial changes its sign due to slow decay of dipolar polarization. Hashimoto *et al.*, (1975) have also observed peaks of opposite polarity in polyethylene electrets and attributed them to hetero- and homo-charge trapping.

Table1: Depolarization kinetic data i.e. activation energy, Relaxatiotimeand Charge Released for pure and various malachite green sensitized PVC samples

Field (kV /cm)	**Temperature ^{o}C**	**Pure PVC (eV)**		**6% M green sensitized PVC (eV)**		**7.5% M green sensitized PVC (eV)**		**10% M green sensitized PVC (eV)**		**15% M green sensitized PVC (eV)**	
20	90	0.384	0.692	0.346	0.671	0.339	0.669	0.329	1.05	0.302	0.987
30		0.380	0.714	0.338	0.669	0.335	0.661	0.326	0.998	0.301	0.980
40		0.379	0.710	0.337	0.667	0.330	0.659	0.323	0.987	0.298	0.979
50		0.372	0.708	0.335	0.657	0.319	0.640	0.321	0.979	0.287	0.972
		Relaxation time in second									
20	90	6.114 x 10^{-12}		6.478 x 10^{-12}		6.875 x 10^{-13}		7.557 x 10^{-13}		8.478 x 10^{-13}	
30		7.135 x10^{-12}		7.244 x 10^{-12}		7.397 x 10^{-13}		8.167 x 10^{-13}		9.246 x 10^{-13}	
40		8.234 x 10^{12}		8.779 x 10^{-12}		9.234 x 10^{-13}		9.439 x 10^{-13}		1.347 x 10^{-13}	
50		8.779 x 10^{-12}		8.997 x 10^{-13}		9.958 x 10^{-13}		1.547 x 10^{-14}		3.578 x 10^{-14}	
		Charge Released in coulamb									
20	90	7.553 x 10^{-7}		1.678 x 10^{-6}		6.789 x 10^{-6}		8.879 x 10^{-6}		2.798 x 10^{-5}	
30		9.997 x10^{-7}		3.789 x 10^{-6}		9.887 x 10^{-6}		3.345 x 10^{-5}		5.478 x 10^{-5}	
40		1.536 x 10^{-6}		6.538 x 10^{-6}		2.431 x 10^{-5}		5.447 x 10^{-5}		9.788 x 10^{-5}	
50		1.987 x 10^{-6}		6.975 x 10^{-6}		2.981 x 10^{-5}		5.998 x 10^{-5}		2.876 x 10^{-4}	

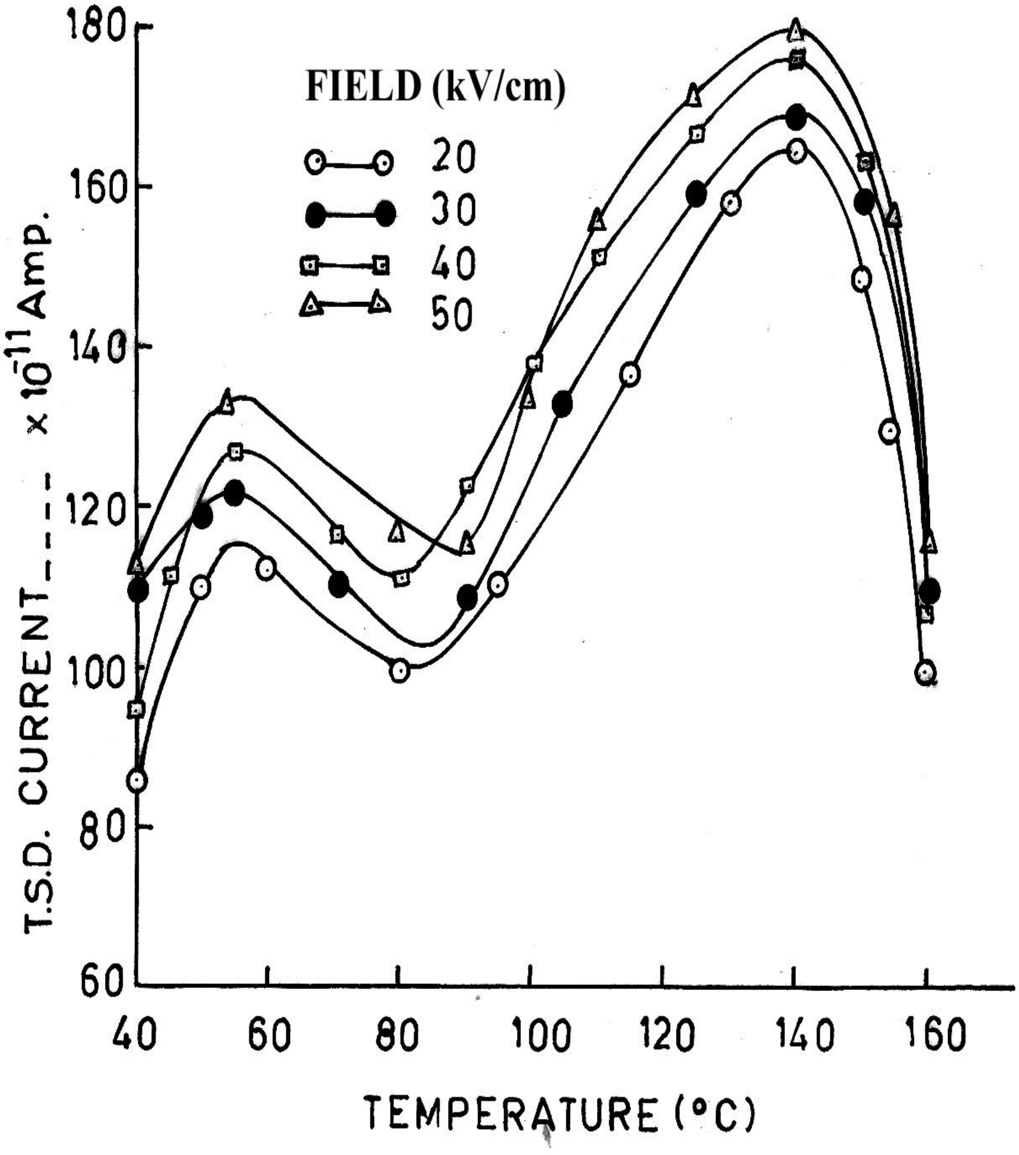

Figure 1: TSDC Thermograms of pure polyvinyl chloride (PVC) samples polarized with various fields at polarizing temperature Tp=90°C

Swan (Swan, 1967) proposed four mechanisms for the charge transfer complex processes involved between the strong electronegative acceptors present in the polymer molecular chain.

(i) The electron may go back to the polymer molecule from which it originates.

(ii) The electron may be retained by the sensitizer and may move as a stable negative ion.

(iii) The electron may transfer from the negative sensitizer into another polymer molecule without motion of the sensitizer molecule and

(iv) A charge exchange may occur between the negative sensitizer ion and a neutral sensitizer molecule.

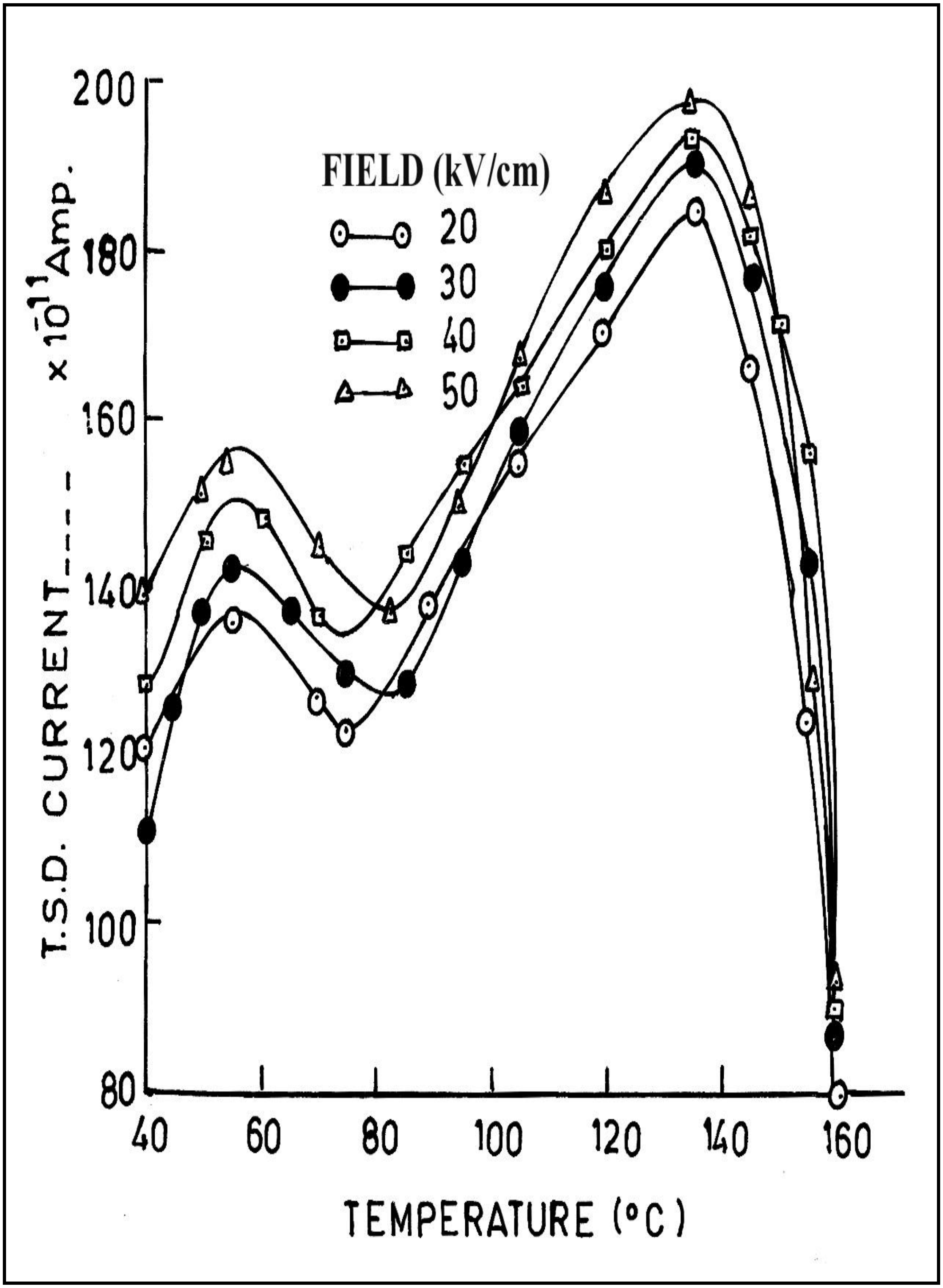

Figure 2:-TSDC Thermograms of 6% malachite green sensitized (PVC) samples polarized with various fields at polarizing temperature Tp=90°C

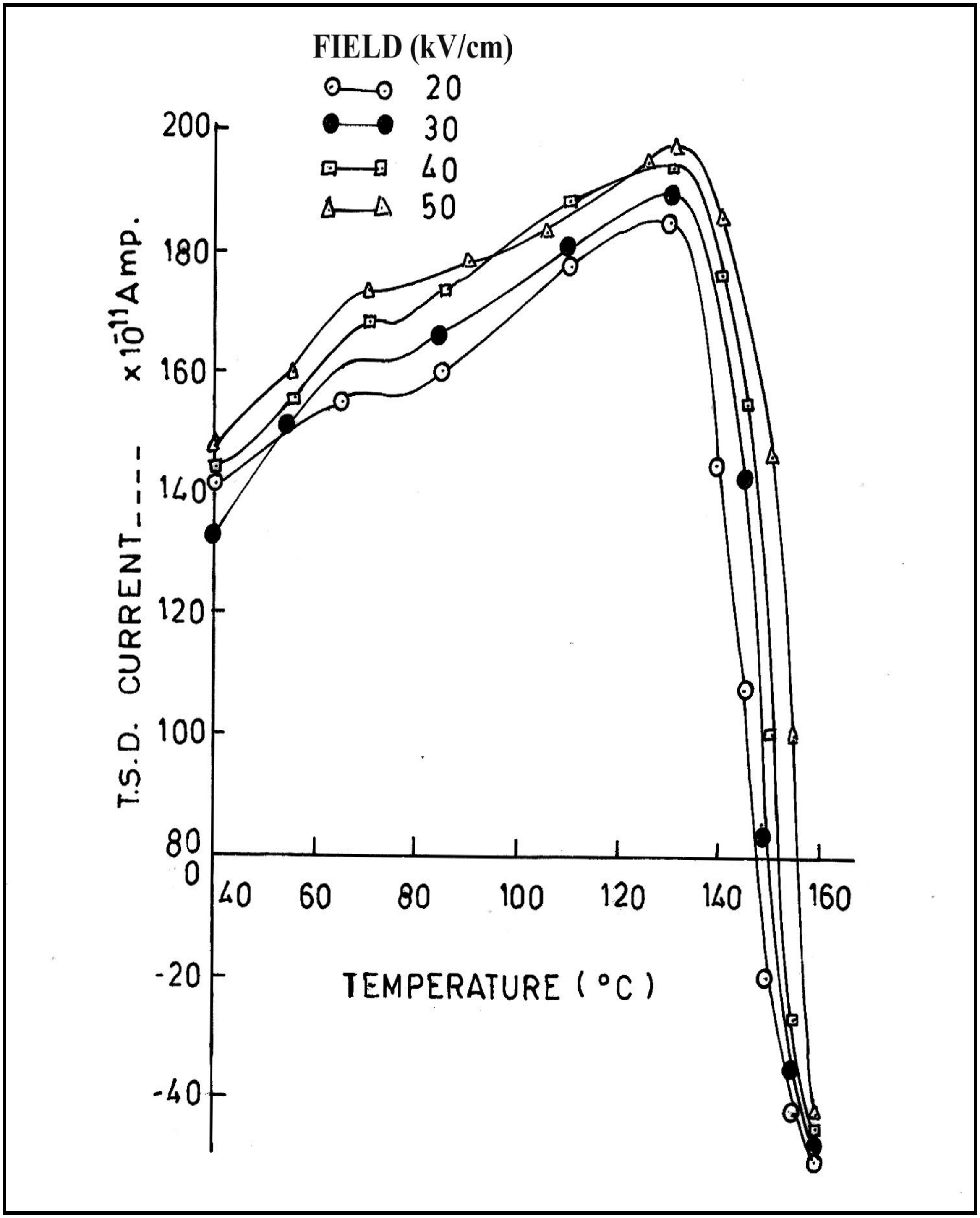

Figure 3: TSDC Thermograms of 7.5% malachite green sensitized (PVC) samples polarized with various fields at polarizing temperature Tp=60°C

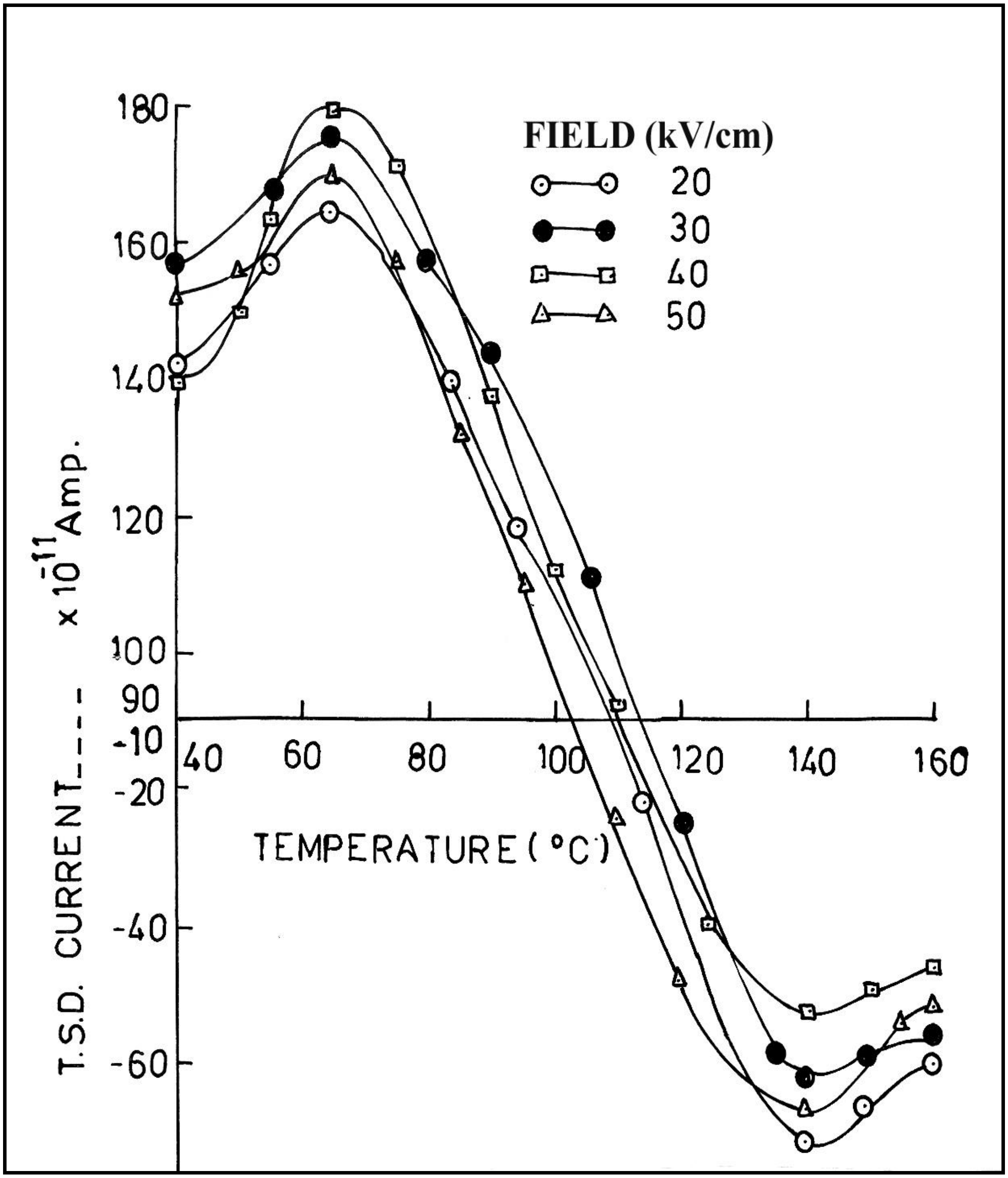

Figure 4: TSDC Thermograms of 10% malachite green sensitized (PVC) samples polarized with various fields at polarizing temperature Tp=90°C

The formation of CTC will result in the reduction of the crystalline amorphous interface and provide conducting paths through the amorphous regions and thus interconnect the crystallites. Due to the reduced barrier at the interfaces the mobility of the dipoles and/or charge carriers will increase.

The increase in depolarization currents and decrease in activation energy of polyvinyl chloride samples on sensitization may be explained by the formation of charge transfer complexes. The addition of sensitizer has a strong effect on the activation energy of the discharge process. The activation energy decreases with sensitizing of malachite green in PVC matrix. The molecular motion obviously assisted by the presence of charge transfer complexes, hence activation energy decreases due to the increase in mobility of charge carriers. The activation energies evaluated in the present investigations are found to be lower than the pure films.

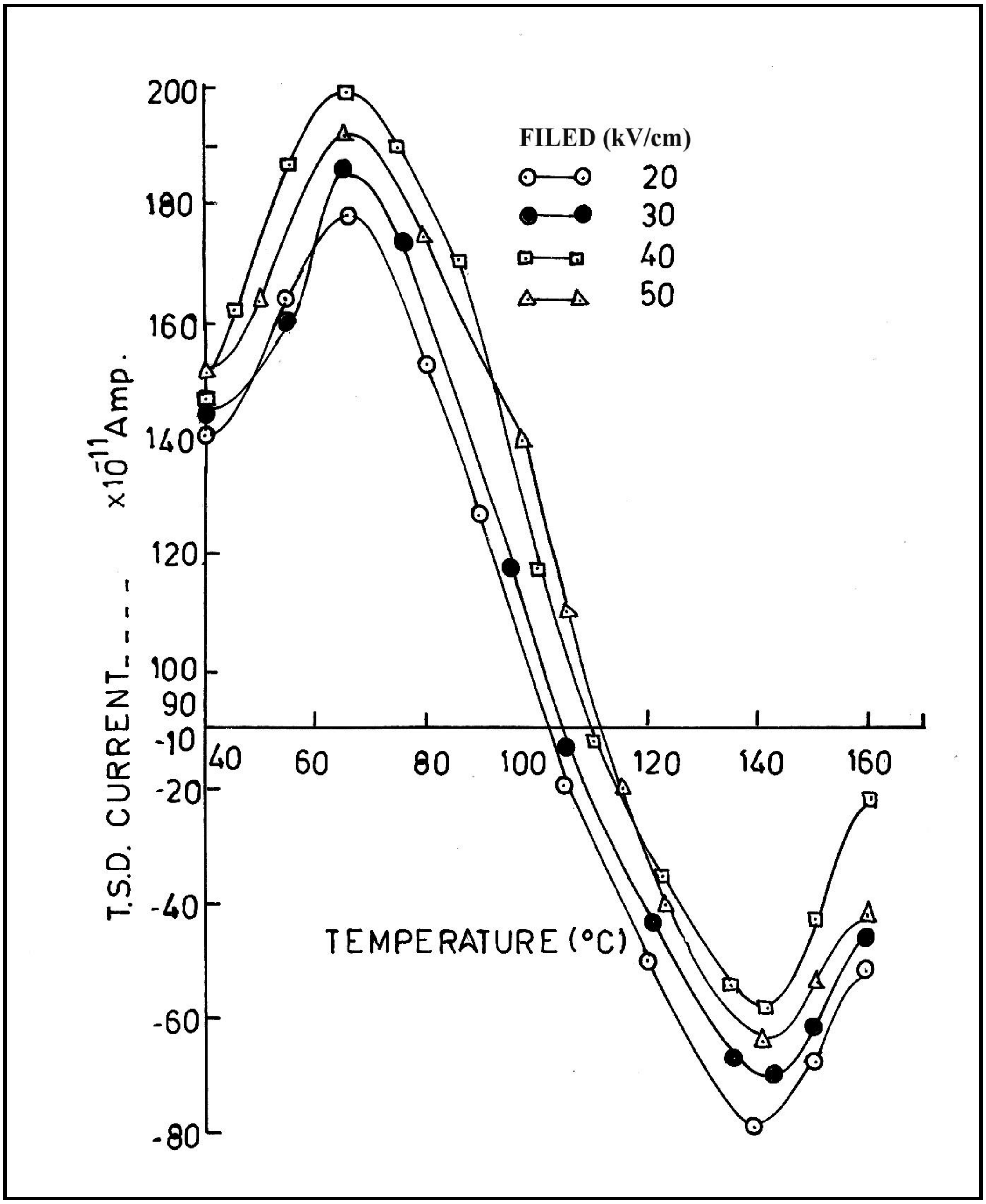

Figure 5: TSDC Thermograms of 15% malachite green sensitized (PVC) samples polarized with various fields at polarizing temperature Tp=105°C

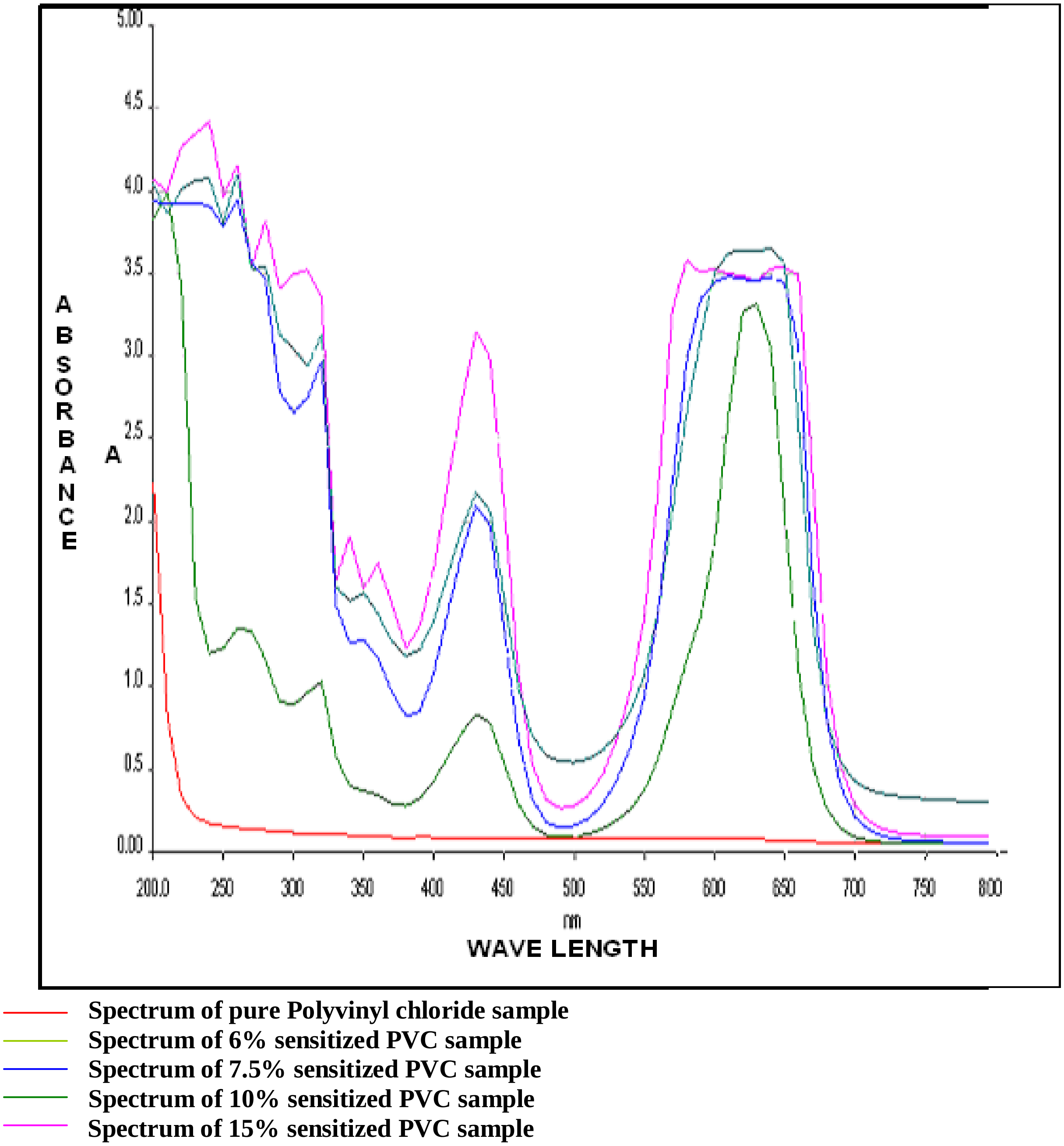

Figure 6: UV Spectra of pure and various malachite green sensitized PVC samples

This indicates that the presence of sensitizer between polar molecules reduces of interaction and these results in a reduction of activation energy. Rastogi and Chopra (1973) observed that the addition of electron acceptor species to polystyrene increases the conductivity of the material and the increased conductivity being associated with mobility has the effect of decreasing the activation energy.

Soon, after the addition of malachite green to PVC solution the colour of solution changes. Malachite green has an amino-group containing an unshared pair of electrons which may be responsible for CTC formation. Malachite green has triphenylmethane structure (Jiska *et al.*, 1981) and it is a cationic dye.

As the dye is cationic, the coloured ion is charged positively (Gutmann and Lyons, 1967). The cationic nature of the dye suggests that it is electron donor dye while PVC is likely to accept electrons. Hence it is very likely that malachite green form donor – acceptor complexes with PVC when mixed it under suitable conditions (Bharadwaj and Khare, 2012; Khare and Singh, 1994).

Ascertaining the existence of a charge transfer complex in polymeric systems is often a difficult task. Many charge transfer complexes are highly coloured and therefore spectral studies are more useful. The formation of charge transfer complexes is evidenced in the UV-visible absorption spectra by the appearance and change in the intensity of absorption bands.

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