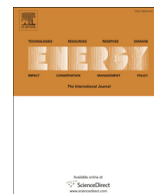




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Investigation of the effects of thermal, oxidative and irradiation treatments on the behaviour of poly-ethylene glycol as a phase change material in thermal energy storage systems

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ABSTRACT

PEG (poly-ethylene glycol) with an average molecular weight of 2000 g/mol has been investigated as a phase change material for thermal energy storage applications. PEG sets were maintained at 80 °C for 861 h in air, nitrogen, and vacuum environment; the samples maintained in vacuum were further treated with air for a period of several weeks. Furthermore, another set of PEG samples was exposed to electron radiation in order to modify some of their polymer properties, such as their melting point T_m , their heat of fusion ΔH_m , their crystallisation temperature T_c , the heat of crystallisation ΔH_c , and their thermal decomposition temperature T_{decomp} . The experiments showed that the presence of oxygen led to the degradation of the polymer and to a slight decrease of its melting temperature, while the treatment with electron radiation reduced polymer's heat of fusion. FTIR (Fourier transform infrared spectroscopy) spectrum analysis showed bands assigned to carbonyl/carboxylate functional groups, indicating the degradation of PEG in the presence of air/oxygen.

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1. Introduction

The storage of thermal energy based on the latent heat of PCMs (phase change materials) has attract the interest of many researchers for more than 80 years [1,27,31,34]. Several suggestions have been made for applications in medicine [2,3] and energy production [4] and especially in the building industry. PEG (poly-ethylene glycol) is one of the most promising PCMs regarding thermal energy storage applications [1–6]. Its advantages include a relatively large heat of fusion [1,6,7], non-corrosiveness [8], non-toxicity [9] and a wide range of phase change temperatures, which can be manipulated by modifying its molecular weight [7,8]. The flexibility of PEG is the main reason why it has been proposed for both heating and cooling systems, based on the appropriate selection of its molecular weight [10,35]. It has also been found that PEG is more reliable in melting–freezing cycles than any other inorganic salt hydrate PCM, as there is no super-cooling phenomenon occurring during its freezing cycles [8,36].

However, the major problem with the use of polymeric materials for thermal energy storage is the decomposition/degradation of polymers. PEG is prone to oxidation, as its lamellar structure chains fold by the free-radicals and as a result the activation energy of hydrogen abstraction and the molecular weight of the polymer are reduced, while oxygenated products with low molecular weights are formed. Consequently, the performance of PEG as a thermal energy storage system reduces with the decrease of PEG's molecular weight [9,11,26].

Beyond thermal oxidation, PEG also subjects to mechanical degradation [12,13]. For instance, without stabilisers, it takes approximately 18 days to completely deteriorate freshly polymerised PEG at ambient temperature [14], but this deterioration may be suppressed by the addition of specific antioxidants [1,6]. Furthermore, it has been observed that PEG also degrades over long periods of time (2–4 years) when stored in large quantities [15].

On the other hand, electron irradiation on hydrophilic polymers like PEG, PVA (poly-vinyl alcohol) and PVP (poly-vinyl pyrrolidone) leads to significant changes of the polymer properties, as crosslinks are created between the polymer chains [16–18], which is part of the process of creating hydrogels [19,20,29].

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Recent studies show that alternative substances may be added to PEG to form composites, in order to increase their stability and/or acquire desired properties [9,21]. These composites are generally more stable and report enhanced features, such as increased temperature ranges [22], or increased conductivity [21,23], but once again at the cost of reducing the overall thermal storage density.

The present work reports the effects of multiple treatments on the behaviour of poly-ethylene glycol with an average molecular weight of 2000 g/mol, to be used as PCM in thermal energy storage systems. The objective is to examine the performance of PEG 2000 under different stimulus, which simulates possible operating conditions. A comparison between untreated samples of PEG and samples treated through heating under nitrogen and oxygen atmospheres, irradiated with electrons and heated in vacuum was conducted; the specimens heated in vacuum were then left in contact with air. The specimens' melting temperature, heat of fusion and weight loss rate were then compared and reported. Infrared analysis was also conducted on all the specimens.

2. Experimental procedure

The reagent grade PEG used was obtained from Fluka, with an average molecular weight of 2000 g/mol. The specimen groups were treated by:

- Oxygen (air) – Glass tubes were filled with PEG powder, and placed in an oven at 80 °C for up to 861 h in air atmosphere.
- Nitrogen – A set of ampoules were filled with PEG powder and nitrogen gas was used to purge the oxygen from the container. Then the ampoules were thermally sealed and placed in an oven at 80 °C for up to 861 h.
- Vacuum – The glass tubes were filled with PEG powder; nitrogen gas was purged into the tubes to remove all the oxygen, then the tubes were sealed with a rubber stopper, evacuated using a syringe, and placed in an oven at 80 °C for different durations up to 861 h. Afterwards, the tubes were left at lab temperature for several weeks. Air and humidity were allowed inside through the rubber stopper.
- Electron Irradiation – The fourth group consisted of poly-ethylene bags filled with PEG powder. Nitrogen gas was purged in order to remove the oxygen from within, and the bags were then sealed. The specimens were exposed to various doses of electron radiation up to 200 kGy. An electron accelerator from Vivarad, France, was used, and 10 mA current was adjusted to determine the dose rate.

2.1. DSC (differential scanning calorimetry)

DSC spectra were recorded using Setaram instrument (type DSC131) in order to determine the melting and the crystallisation temperatures of the samples as well as the heat of fusion and the heat of crystallisation. All samples were tested in aluminium pans at a heating rate of 10 °C/min over a suitable temperature range. The precision of the instrument was ± 0.2 °C, and the experimental errors in the measurements were estimated to be about ± 0.5 °C.

2.2. TGA (thermogravimetric analysis)

Thermal decomposition of PEG was followed up using a thermogravimetric analyser in a Nitrogen (N₂) atmosphere. Dynamic weight loss was recorded using Mettler instrument (TG50) at a heating rate of 10 °C/min. The total experimental error in the determination of the decomposition temperatures was estimated to be not more than ± 3 °C.

2.3. FTIR (Fourier transform infrared spectroscopy) measurements

FTIR spectra were recorded using a Jasco spectrometer (FT/IR-4200 type A).

3. Results and discussion

3.1. DSC measurements

DSC thermograms for the heating and cooling processes of a fresh PEG sample were recorded in order to simulate the charging and discharging processes which the polymer would experience throughout its working life. The tests were done in nitrogen atmosphere and repeated ten times using the same heating/cooling rate of 10 °C/min. The thermograms for these tests are represented in Fig. 1, in which one can observe that PEG melts and crystallises with an expected behaviour, indicating that it can be used for energy storage systems.

The enthalpies and temperatures for the heating and cooling tests have been determined from the DSC thermograms and are represented in Fig. 2. It can be seen that there is no significant change in the melting enthalpy and temperature of the used PEG during the charging/discharging processes. Thus, it can be concluded that this polymer is suitable for energy storage applications in nitrogen atmosphere.

Changes in the melting temperature and heat of fusion are of great interest from the view point of thermal energy storage. Fig. 3 shows the DSC thermograms of PEG treated in the following manners: thermally treated in nitrogen atmosphere for 861 h at 80 °C, exposed to 200 kGy of electron radiation, heated in oxygen atmosphere for 861 h at 80 °C, heated in vacuum for 861 h at 80 °C and left for four weeks at ambient temperature in contact with air (through leak).

The PEG that was annealed in the presence of nitrogen was characterised by a very similar melting temperature and heat of fusion values compared to the original untreated PEG. However, the PEG specimen annealed in the presence of air/oxygen, the specimen heated in vacuum, and the specimen exposed to 100 kGy of electron radiation, all showed lower melting temperatures and heat of fusion rates than the original untreated PEG. These reductions could be caused by the decrease of the molecular weight of the polymer and the formation of volatile compounds during the degradation process [1].

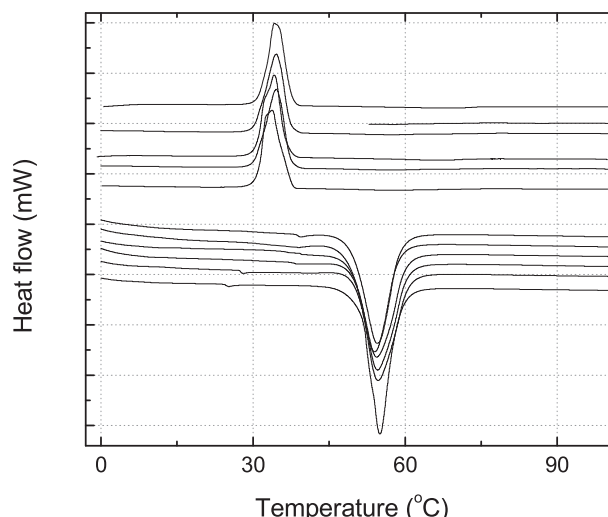


Fig. 1. DSC thermograms of fresh PEG heated and cooled ten times (10 cycles).

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