



Influence of cellulose content on thermal properties of poly(lactic) acid/cellulose and low-density polyethylene/cellulose composites

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Abstract. Crystallization behaviour of polylactic acid/cellulose (PLA/CELL) and linear low-density polyethylene/cellulose (LDPE/CELL) composites was studied by differential scanning calorimetry (DSC) and polarized light microscopy equipped with hot-stage. The effect of addition of cellulose on thermal properties of PLA/CELL and LDPE/CELL composites was considered. The DSC experiments were performed at different scanning rates.

For pure PLA the crystallization peak at $T = 98.8^\circ\text{C}$ was observed in DSC scans at $20^\circ\text{C}/\text{min}$ with the corresponding melting peak at 110.1°C . With addition of cellulose no crystallization peaks and melting peaks related to them were found. For all samples cold crystallization peaks at around 130°C and corresponding melting peaks at $152\text{--}153^\circ\text{C}$ were observed in endotherms. Cold crystallinity is strongly affected by cellulose content: the higher the cellulose content, the higher is the cold crystallinity of the samples. At the same time overall crystallinity for all PLA/cellulose composites is negligible contrary to pure PLA, which is slightly crystalline.

Unlike PLA, LDPE and its composites have no cold crystallization, which is a usual behaviour for LDPE materials. A general crystallization/melting behaviour of LDPE/cellulose composites is identical to that of pure LDPE. However, the crystallinity of LDPE/composites decreases with increasing cellulose content.

Key words: calorimetry, light microscopy, composites, cellulose, polylactide, low-density polyethylene.

INTRODUCTION

Over the past decade, much attention has been focused on biodegradable and biocompatible polymers. Natural polymers, biopolymers, and synthetic polymers based on annually renewable resources are the basis for the twenty-first-century portfolio of sustainable, eco-efficient plastics. In this direction, polylactide (PLA) is one of the most promising candidates. Polylactide has good mechanical properties, thermal plasticity, and is readily fabricated, thus being a promising polymer for various end-use applications. Along with other bio-sourced materials, PLA will gradually replace the currently existing family of oil-based polyolefin polymers (polyethylene, polypropylene, polyethylene terephthalate) as they become cost- and performance-wise competitive [1,2]. Polyolefins are hardly biodegradable, therefore addition of cellulose as filler can improve that property [3].

Most sustainable plastics cannot compete economically with conventional petroleum-derived plastics in their present state. Economically favourable composites, therefore, are expected to be made from costly sustainable plastics in combination with inexpensive natural reinforcement fibres.

Cellulosic materials are the most abundant form of biomass and the form most likely to be used as reinforcement fibres, not only for ecological and economic reasons, but also because of their high mechanical and thermal performance. To utilize and design materials successfully for industrial applications, it is imperative to determine first the material properties that will affect performance [4].

The thermal behaviour of PLA and low-density polyethylene (LDPE) cellulose-based composites has attracted much attention and has been well studied by dynamic mechanical thermal analysis and thermogravimetric analysis [2,5–13]. However, differential scanning calorimetry (DSC) studies of PLA and LDPE composites have been poorly presented in literature. The

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most part of these studies has been performed in the isothermal mode [2,6–9]. Nevertheless, non-isothermal crystallization is also important from both the scientific and commercial viewpoints, because polymer processing is mainly conducted under non-isothermal conditions and this, in turn, affects mechanical properties of the end-product [6].

The non-isothermal behaviour of two-component composites of cellulose with pure PLA or LDPE has not been well investigated [7,14,15]. Therefore, the aim of this work was to study the influence of added pure cellulose on the non-isothermal behaviour of PLA and LDPE by DSC. We have manufactured and studied two-component composites to have a comparison reference for future research of multi-component composite materials containing compatibilizer/coupling agents.

EXPERIMENTAL PART

Materials

Two types of composites were investigated. First, composites compounded of PLA and cellulose fibres, which were abbreviated as PLA/CELL- $n\%$, where $n = 0, 2, 5$, and 10 means the cellulosic weight content in composites. The content of cellulose is expressed in weight per cent to the weight of composite. The second type contained LDPE and cellulose, abbreviated as LDPE/CELL- $n\%$, where $n = 0, 2, 5$, and 10. The LDPE powder was obtained from Borealis Polymers Co (product name: PE FA3220). NatureWorks LLC supplied poly(lactic) acid granules (PLA Polymer 4042D). It has been found in work [1] that this polymer consists of L-isomer 92% and D-isomer 8%. PLA 4042D has an overall degree of crystallinity 2.0%, which can be explained by a high D-isomer content of 8%. Molar masses (weight-averaged M_w) were estimated by rheology using a single exponential model for molecular weight distribution determination with the rheometer Anton Paar Physica MCR 501. Apparent M_w of LDPE and PLA are 400 kg/mol and 77 kg/mol, respectively. Acid-free α -cellulose (chemically clean, no lignin) with a maximum length of $60 \pm 10 \mu\text{m}$ and width $5 \pm 2 \mu\text{m}$ were supplied by Hahnemühle FINEART. Cellulose dimensions were measured by the polarized light optical microscope Zeiss Axioskop-2.

Composite processing

First, cellulose and polymer powders were prepared. Cellulose sheets were crushed into pieces and then ground to a powder using the cutting mill Retsch SM 100. Polylactide and LDPE granules were then pulverized in the Retsch cutting mill and then PLA powder was dried in an oven at 105 °C for 24 h. Composites of LDPE and PLA with cellulose were prepared by addition of cellulosic powder to matrix polymer.

Polymer/cellulose mixture of different compositions was mechanically stirred for 10 min at 60 rpm in a Labor mechanical mixer. Then polymer/cellulose samples were prepared by compounding on a twin-screw extruder (Brabender Plasti-Corder PLE 651 with a twin-screw compounder DSK 42/7): LDPE and PLA powders pre-blended with cellulose are fed to the extruder at controlled feed-rates. After this the powders were mixed and melted in the extruder via the rotating screws to produce the final molten product that is extruded in the end as strands through a hole in a die plate. There are four temperature zones in the extruder from hopper to die; melting begins from the second zone. The following temperatures were set at respective zones: 150 °C near the feeder, 175 and 190 °C in the middle zones, and 210 °C at the die for LDPE composites. For PLA composites different temperatures in four zones were used: 160, 170, 180, and 180 °C. Both pure LDPE and PLA have undergone the same extrusion procedure as their composites. The molten strand is then cooled by ventilators and chopped into pellets, and then left to cool for 1 h.

We did not use any antioxidants because, as it is known from [16], for PLA strong oxidation at temperatures lower than 200 °C does not occur: thermal degradation mainly occurs due to the chain splitting and not hydrolysis [17]. Low-density polyethylene itself is thermally rather stable at the used temperatures.

Measurements

Differential scanning calorimetry

The Perkin Elmer differential scanning calorimeter DSC-7 was used for thermal analysis at scanning rates of 5, 20, 50, and 100 °C/min. The instruments were calibrated using indium and tin at all applied heating rates. Nitrogen was used as furnace purge gas. Differential scanning calorimetry (DSC) measurements were calibrated for a sample mass of 3 mg. In all DSC experiments the sample mass of $3.00 \pm 0.03 \text{ mg}$ was held constant. Flat samples were packed into aluminium foil to maximize the thermal contact between the sample and the calorimetric furnace. During the measurement, the sample was first held at 190 °C for 5 min to delete its thermal history. Then it was cooled to 0 °C at a definite scanning rate and an exotherm was obtained. After that the sample was held at zero temperature for 5 min and then heated to 190 °C at the same rate to get an endotherm. Both melting and crystallization parameters were obtained from the exotherms and endotherms.

The degree of overall and cold crystallinity of each sample was calculated using the relationship [7]

$$X_c (\% \text{ crystallinity}) = \frac{\Delta H_m}{\Delta H_m^0} \times \frac{100}{w}, \quad (1)$$

where ΔH_m is the enthalpy of melting, ΔH_m^0 is the enthalpy of melting for a 100% crystalline PLA sample ($\Delta H_m^0 = 93$ J/g) [18] or 100% crystalline LDPE sample ($\Delta H_m^0 = 293$ J/g) [19], and w is the weight fraction of PLA and LDPE in the composite.

Polarized light microscopy with hot-stage

The polarized light microscopy (PLM) pictures of all samples were obtained by the polarized light optical microscope Zeiss Axioskop-2 with total magnification of $100\times$ ($10\times$ objective and $10\times$ eyepiece), equipped with a hot stage Mettler FP 80. Test specimens for hot-stage microscopy were prepared by pressing the composite pellets between two cover glasses at 200°C , using a hot plate to produce a very thin composite film with an average thickness of $20\ \mu\text{m}$ measured by the thickness meter Sony DZ521. Samples were first heated from 20 to 190°C at $20^\circ\text{C}/\text{min}$, held at this temperature during 5 min, and then cooled at the same speed to 25°C and heated again. Snapshots at different temperatures during the second heating were recorded.

RESULTS AND DISCUSSION

Figure 1 shows the typical non-isothermal DSC thermograms of pure PLA and PLA/cellulose composites,

recorded at $20^\circ\text{C}/\text{min}$. The pure PLA is characterized by glass transition temperature T_g (marked in Fig. 1 as G) at 58.0°C , which agrees with literature data [7], the crystallization peak at $T_c = 98.8^\circ\text{C}$ (Fig. 1a, marked with C), and the corresponding melting peak T_m at 110.1°C (see Fig. 1b, marked with M). In-depth analysis of these thermograms revealed a small endothermic peak around 60°C , typically attributed to stress relaxation on heating [20]: after fast cooling to a temperature below T_g , the material goes into the glassy state and tends to decrease its excess enthalpy moving towards the equilibrium glassy state. This excess enthalpy is recovered at the glass transition region during the subsequent heating process.

Addition of cellulose results in diminishing the crystallization peak on the cooling curves and as a consequence no corresponding melting peak on the heating curves has been found. The degree of crystallinity of pure PLA was only about 2% and addition of cellulose makes composites fully amorphous. This implies that addition of cellulose produces a negative effect into the overall crystallinity of composites. A similar behaviour was observed in work [7] for PLA composites: the degree of crystallinity of composites with different cellulose reinforcements decreased compared to pure PLA.

As can be seen from the melting curves in Fig. 1, all PLA composites, including the pure polymer, have cold crystallization peaks (T_{cc}) located at 130 – 134°C (marked with Cc in Fig. 1b) and subsequent melting peaks (T_{mc})

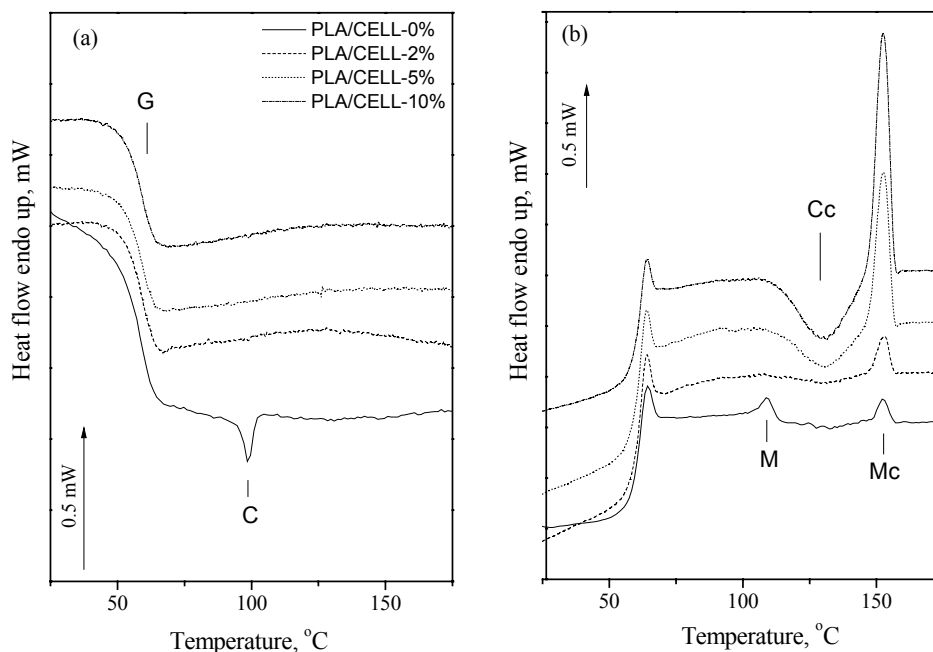


Fig. 1. DSC exotherms (a) and endotherms (b) of PLA and PLA/CELL composite materials: 2nd heating, heating rate $20^\circ\text{C}/\text{min}$. The arrows indicate the glass transition (G), the cold crystallization (Cc), and the melting related to the cold crystallization peak (Mc). There is an additional melting peak (M), related to the crystallization peak C of pure PLA.

at 152–153 °C (marked with Mc in Fig. 1b). However, it should be noted here that the PLA/CELL composites are assumed to be amorphous since cold crystallization enthalpy ΔH_{cc} and melting enthalpy ΔH_{mc} values are almost the same. Cold crystallization peaks indicate regions of realignment and transition from amorphous to crystalline phases. Figure 2 shows an evolution of cold crystallinity in PLA/CELL composites observed by PLM. Temperatures of 25, 135, and 155 °C were chosen for snapshots. It is clearly seen that the amount of crystallites increases gradually in the range of 130–140 °C during the non-isothermal process and, finally, the formed crystals melt at 155 °C.

From DSC data of PLA/CELL composites it was found that T_{cc} decreases with cellulose content downwards from 134 °C for pure PLA to 130 °C for PLA/CELL-10%. At the same time, the values of ΔH_{mc} and ΔH_{cc} of PLA/CELL samples increased with cellulose content. This behaviour indicates that cellulose induces crystal nucleation of the PLA polymer [21]. Cellulose appeared to facilitate nucleation and subsequent crystallization of PLA rather effectively in amorphous composites. Similar results were obtained by different authors in the case of PLA/talc and PLA/kenaf composites [22,23]. The enhancement in melting and cold crystallization enthalpies are opposed to several studies reported on PLA composites reinforced in the presence of a coupling agent [23,24].

Further, values of ΔH_{mc} and ΔH_{cc} are dependent on cooling/heating rates. Figure 3 shows DSC curves for PLA/CELL-10% at different heating rates. The heating rate dependence on glass transition temperature T_g is an experimentally observed fact [25,26]: if the heating or cooling rate is increased in magnitude, then the relaxation time becomes smaller and the transition region is shifted to a higher temperature. In the present study, too, T_g increases with increasing heating rate.

It is more interesting that peaks related to cold crystallization are dependent on the heating rate. With

increasing heating rate, T_{cc} shifts gradually to a higher temperature owing to the thermal delay [27]. It is notable that as the heating rate rises above 20 °C/min, both ΔH_{mc} and ΔH_{cc} decrease significantly and peaks are hardly observed. When scanned at 5 °C/min, two endothermic peaks appeared. Apparently, the amorphous PLA chain does not have enough time to well self-adjust with increasing heating rate. This results in the decrease in cold crystallization, and the multi-melting behaviour, which can be attributed to the simultaneous occurrence of melting of thinner lamellae and recrystallization. Such a behaviour is very common for most of the composites based on PLA, for example PLA/flax [21], PLA/nucleating agent $\text{SiO}_2(\text{TiO}_2)$ [2], PLA/clay [27], etc., and is probably attributed to the PLA nature. However, the filler can affect the cold crystallization process of PLA in one way or another. Remarkably, the cold crystallization process is dependent only on the heating rate and independent of the cooling rate during the cooling process prior to the 2nd heating. Indeed, as can be seen from the inset to Fig. 3, T_{cc} , ΔH_{mc} , and ΔH_{cc} are identical for all used cooling rates if the heating rate is the same.

The thermal behaviour of LDPE-based composites differs strongly from that of PLA/CELL composites. First of all, LDPE has no cold crystallization regions on the heating curves, which is usual for LDPE materials [19].

Figure 4 shows a non-isothermal cooling and heating process of LDPE and their cellulose composites. Exotherms are characterized by the presence of two crystallization peaks at 61–62 °C and 98 °C, whereas the endotherm has only one broad melting peak at 110–111 °C. However, such a thermal behaviour is expected for LDPE material [19].

Addition of cellulose does not affect any crystallization and melting temperatures. As is known, the melting point of a semicrystalline polymer is determined by the size and perfection of its crystals (i.e. by the lamellar

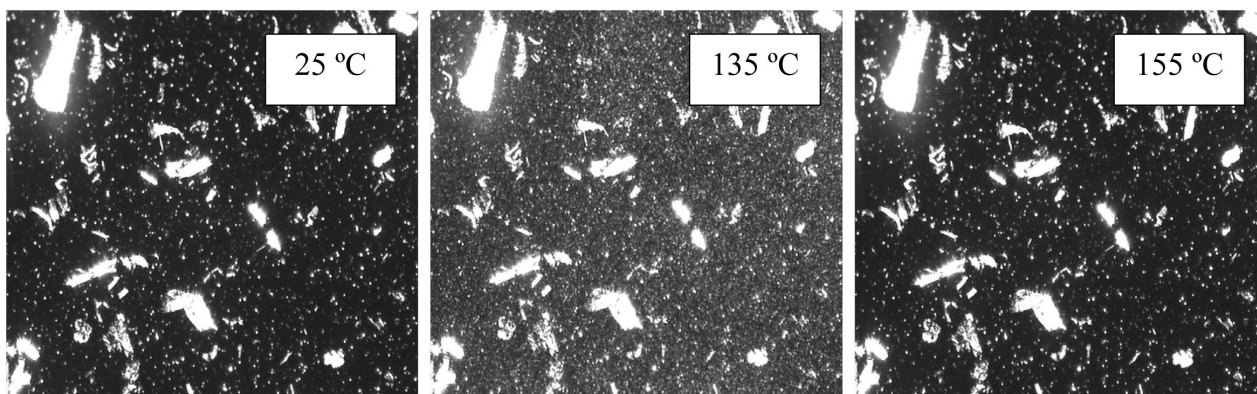


Fig. 2. PLM pictures of PLA/CELL-5% snapped at temperatures 25 °C, $T_{cc} \cong 135$ °C, and 155 °C during heating at 20 °C/min.

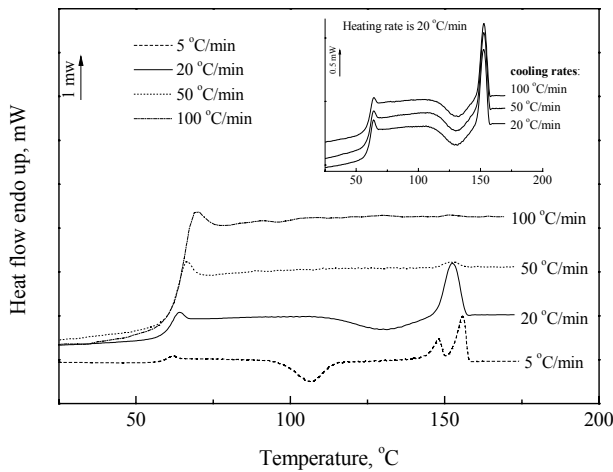


Fig. 3. DSC melting curves of PLA/CELL-10% at different heating rates. The inset shows endotherms for the same sample at different cooling rates, but at the same heating rate of 20 °C/min.

thickness) [19]. Taking into account that the crystallization and melting temperatures of the LDPE and LDPE/CELL samples do not change with cellulose content, it can be concluded that the size of LDPE crystallites keeps the same value.

On the contrary, both ΔH_m and the degree of crystallinity were affected by the presence of the filler. For all systems, ΔH_m decreases monotonously as the filler content in the composite increases, as expected,

because of the decrease in the matrix material in composites. However, the degree of crystallinity, calculated by eq. (1) and accounted for the effective matrix amount in the composites, also decreases with cellulose content from 42% for pure LDPE to 38% for LDPE/CELL-10%. Decrease in crystallinity is clearly observed in PLM pictures presented in Fig. 5. The filler seems to hinder the crystallization of the surrounding polymeric matrix and formation of crystalline domains.

CONCLUSIONS

The non-isothermal crystallization and melting behaviour of PLA/CELL and LDPE/CELL composites prepared by compounding in a twin-screw extruder was studied using DSC and polarized light microscopy with a hot-stage.

Thermal analysis showed an increase in cold-crystallization enthalpy in PLA composites, indicating that cellulose enhances nucleation and crystal growth.

Addition of cellulose has no effect on glass transition temperature T_g of PLA/CELL composites. However, the heating rate has an impact on T_g and T_{cc} , showing increasing values with a higher heating rate. At the same time, the heating rate affects ΔH_{mc} and ΔH_{cc} , resulting in the disappearance of the cold-crystallization process under applied high heating rates above 50 °C/min. Obviously a high heating rate does not allow enough time for PLA chains to rearrange, resulting in the decrease in cold crystallization.

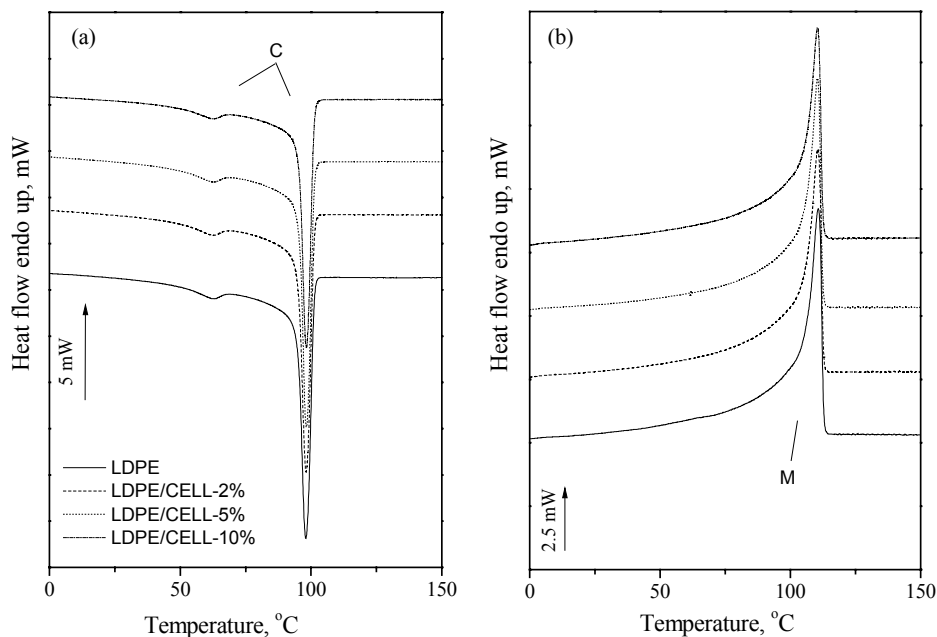


Fig. 4. DSC exotherms (a) and endotherms (b) of LDPE and its composites with cellulose: 2nd heating, heating rate 20 °C/min. The arrows indicate the crystallization (C) and the melting (M) related to crystallization peaks.

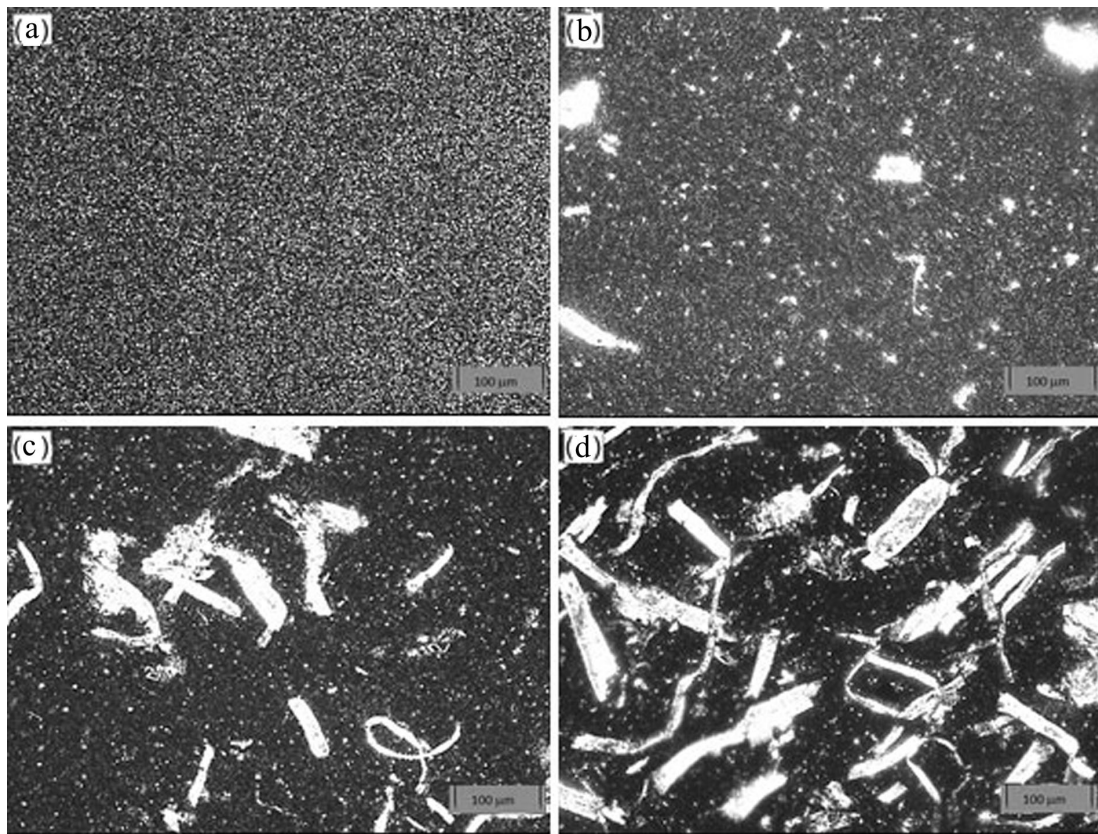


Fig. 5. PLM pictures of LDPE and its composites taken at 25 °C: (a) pure LDPE, (b) LDPE/CELL-2%, (c) LDPE/CELL-5%, (d) LDPE/CELL-10%.

The thermal behaviour of LDPE/CELL composites in general is similar to that of pure LDPE. Crystallization and melting temperatures of composites and LDPE are the same and do not change with an increasing cellulose content. Incorporation of cellulose to LDPE reduces only the overall crystallinity of composites. Probably, cellulose hinders the migration and diffusion of LDPE chains resulting in a weaker crystallization process compared to neat LDPE.

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Tselluloosi sisalduse mõju polülaktiidi/tselluloosi ja madaltiheda polüetüleeni/tselluloosi komposiitide termilistele omadustele

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Käesolevas töös uuriti polülaktiidi/tselluloosi (PLA/CELL) ja madaltiheda polüetüleeni/tselluloosi (LDPE/CELL) komposiitide termilisi omadusi, kasutades diferentsiaalset skaneerivat kalorimeetriat (DSC) ning kuumaluse valgusmikroskoopi. Komposiidid valmistati kompaundimise teel, kasutades kaheteolist ekstruderit. DSC ja kuumaluse valgusmikroskoopia uuringud viidi läbi mitteisotermilises režiimis kuumutus- või jahutuskiirusega 20°C/min.

Tselluloosi lisamine muudab PLA/CELL-i komposiidid amorfseteks. Siiski on PLA ja selle komposiitide isoleerimiseks omaduseks külmkristalliseerumine kuumutusprotsessis. Kõikidel PLA/CELL-i komposiitide DSC endotermidel esinesid külmkristalliseerumise haripunktid vahemikus 130–134°C ja nendele vastavad sulamisharipunktid temperatuuril 152–153°C. Summaarselt on PLA/CELL-i komposiidid siiski amorfset, sest külmkristalliseerumise entalpia ΔH_{cc} ja sulamise entalpia ΔH_{mc} on peaaegu võrdsed. Lisaks vähenevad ΔH_{mc} ja ΔH_{cc} oluliselt kuumutuskiiruse suurenemisega üle 20°C/min ning seetõttu on haripunkte raske tuvastada. Ilmselt ei ole amorfsetel PLA ahelatel kuumutuskiiruse suurenemisega ümberkordistamiseks piisavalt aega ja selle tulemusena väheneb külmkristalliseerumise aste. PLA/CELL-i (tselluloosi sisaldus 10%) sulamisprotsessi mikroskoopilisel vaatlusel tuvastati kristalliliste superstruktuuride moodustumine temperatuurivahemikus 130–140°C ja nende täielik sulamine temperatuuril 155°C.

DSC uuringute põhjal määrati kindlaks, et tselluloosi lisamine ei mõjuta PLA klaasistumistemperatuuri, kuigi kuumutuskiiruse kasv põhjustab selle tõusu.

LDPE ja selle komposiitide käitumine erineb tugevalt PLA/CELL-i komposiitidest. LDPE-l puudub külm-kristalliseerumine. LDPE/CELL-i komposiitide DSC eksotermidel esineb kaks haripunkti: 61–62 °C ja 98 °C. Nendele vastab üks lai LDPE-le iseloomulik sulamisharipunkt temperatuuril 110–111 °C. Tulemused näitavad, et lisatud tselluloos ei mõjuta komposiitide kristalliseerumis- ja sulamistemperatuure. Samas alandab tselluloosi lisamine komposiitide kristallisatsiooniatet: 42 protsendilt puhta LDPE puhul kuni 38 protsendini LDPE/CELL-10% komposiidi puhul. Mikroskoopiapiltidel on selgelt näha tselluloosikoguse kasvust tingitud kristalliliste superstruktuuride arvu vähenemine komposiitides. Võib oletada, et tselluloos takistab LDPE makromolekulide migratsiooni ja difusiooni kristallisatsioonifronti. Seda saab ka tõlgendada kui nõrka segunevust LDPE/CELL-i segudes.

Kokkuvõtteks võib järeldada, et kuigi PLA ja LDPE leiavad kasutust sarnastes rakendustes, on termilistest omadustest lähtuvalt tegu väga erinevate materjalidega.