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LMPP Effects on Morphology, Crystallization, Thermal and Mechanical Properties of iPP/LMPP Blend Fibres

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Abstract

The thermal properties and morphological characterisation of isotactic polypropylene (iPP) homopolymer and its blends with low molecular low modulus polypropylene (LMPP) were studied. Firstly blends were prepared with variant LMPP contents, and their properties were characterised using SEM, DSC, XRD, and DMA. Later the mechanical properties of iPP/LMPP blend fibres were investigated. SEM results showed that the iPP/LMPP blends produced smoother surfaces when the LMPP content was increased, as well as the miscibility. All the T_g values with different LMPP percentages were in-between pure iPP and LMPP. The XRD results indicated the LMPP percentage decreased along with the degree of crystallinity of the iPP/LMPP blends (5% to 15%), which increased and then decreased as compared to pure iPP. The elongation at break increased when the LMPP content increased, with the maximum elongation at break of the LMPP 25% blend reaching 12.95%, which showed great stretch-ability, whereas the elastic modulus of iPP/LMPP blends decreased.

Key words: polypropylene, iPP/LMPP blends, morphology, crystallinity, low molecular low modulus polypropylene.

Many investigations have shown that the blending of two or more polymers is economical and more efficient than synthesising a new material [3]. The mechanical properties of commercial polypropylene (PP) homopolymers depend on the processing conditions, crystallisation and rheological behaviour, which are dependent on the molecular structure, molar mass distribution and stereoregularity or the molecular chain structure [4]. The molecular weight (MW) also plays an important role in defining the morphology and crystallisation of the blends.

Moreover, in the last few decades, PP blending with other polymers has received vast theoretical and experimental investigation as the melting, morphology, crystallinity and crystallization behaviour are mainly dependent on the blended components. Morphological complications were reported when it was blended with different polymers, specifically containing crystal-crystal interactions and amorphous-amorphous interactions, with an unclear understanding of their miscibility [5]. Studies were performed on crystalline/crystalline polymer blends, for instance, linear low-density polyethylene (LLDPE), modified polypropylene [6], and stereo-block polypropylene/isotactic polypropylene blends [7-10]. Detailed studies can be found from the literature related to iPP and atactic polypropylene (aPP) blends covering crystallisation temperature and blend compositions [11-13]. However, the aPP used for blends shows low tacticity and is energy

consuming. Moreover morphological characterisation is still found to be complicated, since liquid-liquid phase separation and liquid-solid de-mixing of the blend components are difficult to examine in their crystallisation phase. Even with a successful blending of iPP with other polymers, there is no certainty of having potent blend fibres, for instance isotactic iPP or metallocene (mPP) requires a certain amount of other (polymer) additives to spin blend fibres. Some researchers found it impossible to spin modified fibres with mPP and an additive such as co-polyamide A, and possibly needed other additives in their blends [14]. Imparting functionality to PP fibres or non-woven fabric with advanced materials, such as metal oxides, nanoparticles etc., to improve antimicrobial and mechanical properties has been reported [15, 16], for example bi-layers of PP. However, it is difficult to distinguish certain properties of the blends, since very little has been known and done on possible blends with other polymers.

Low molecular, low modulus polypropylene (LMPP) is a newly modified polypropylene developed by Idemitsu, Japan, which enables to generate nonwovens characterised by softness and elastic derived from a polyolefin. It can also be used as a base polymer for hot melt adhesives (HMA), hygiene textile products and diapers. The company has proposed three methods of the production of LMPP, which is actually a low molecular, low modulus polypropylene. The meth-

Introduction

The isotactic polypropylene (iPP) polymer is the most widely used polymer among other polymeric materials, due to its low price and impartial mechanical properties, although produced with one major drawback – its brittleness at low temperatures. This is generally overcome by blending with elastomeric polymer materials to improve the impact resistance and extending its applications [1, 2].

ods proposed are as follows: i) blending with elastomers, ii) copolymerisation with other monomers e.g. ethylene, and iii) controlling stereo-regularity [17,18]. LMPP, which is a low tacticity polypropylene, is receiving great attention due to its great spinability and elongation.

The main purpose of this research was to evaluate the morphological properties of iPP and LMPP blends in order to improve their mechanical properties, such as toughness, elongation and spinability. The potential emphasis was on applications of these blend materials in the nonwoven manufacturing (spun-bond, melt blown and spun-lace) and recycling industries. The LMPP contents studied were at 5%, 10%, 15%, 20% and 25% with iPP. The morphology, crystallisation and melting behaviours of LMPP blends with iPP were evaluated, having different molecular weight and molecular weight distribution. Morphological properties of the iPP/LMPP blends were investigated by DSC, SEM, DMA, and XRD, and later the mechanical properties with iPP/LMPP blend fibres were measured.

Experimental

Materials

Isotactic polypropylene (iPP), s2040 (Ziegler–Natta) with an average molecular weight of 2.0×10^5 g/mol was provided by SECCO Petrochemical limited, China. The low molecular weight and low modulus polypropylene (LMPP s901) were provided by Idemitsu Kosan Co, Ltd. Japan, with an average molecular weight of 1.3×10^5 g/mol. The blend mixing ratios of iPP/LMPP by (wt/wt) were as follows: 95/5, 90/10, 85/15, 80/20, and 75/25, and are defined as 5%, 10%, 15%, 20%, and 25%. Morphological effects of the iPP/LMPP blends were investigated by comparing 100% PP and 100% LMPP.

Blending and specimen preparation

Mechanical blends of iPP/LMPP at different compositions were prepared with the help of a mechanical blender, made by Giant Co, Ltd. China, mixed for 2 minutes at room temperature. Melt blended samples of the homopolymers with various compositions were prepared by means of a twin-screw extruder (TSE-30A Ruiya extrusion system Co, Ltd., Nanjing, China) with an L/D = 40, the processing speed of which was 60 rpm at 210 °C. The samples were then cut into pellet form after drawing.

Morphological characterisation

Morphological investigation of the blend samples was carried out with a JSM-5610LV scanning electron microscope (JEOL Company, Japan). All the samples after melt-mixing were cryo-fractured in liquid nitrogen for 15 minutes. Samples were coated with gold prior to scanning. The fractured surface was investigated with secondary electrons of 5kv to get SEM images of all the samples.

Dynamic mechanical analysis

For evaluation of the glass transition (T_g) of iPP, LMPP and blends of iPP were used for dynamic mechanical analysis (DMA), using a Q800, TA Company, USA. Samples were cut from films of the iPP blends after compression into a material rectangular shape of 11.5 mm × 6.5 mm × 0.3 mm size. The method of testing was frequency sweep, and the temperature range over which the glass transitions were measured was at -30 °C to 100 °C. The ramp was 3.00 °C/min at 100 °C/min. The tests were conducted at a frequency of 1Hz.

Thermal behaviour of iPP/LMPP blend samples

iPP and LMPP blend samples were measured for crystallization and melting behaviour with a Perkin Elmer DSC 8000 differential scanning calorimeter (Perkin Elmer USA), having an intercooler lowermost temperature of about -90 °C. The temperature of samples was calibrated using pure Indium ($T_m = 156.6$ °C). Samples weighing about 5 mg were cut from the blended specimen and loaded into the sample pan, under a nitrogen atmosphere from room temperature to 200 °C. The first heating rate used was 10 °C/min, isothermal holding at 200 °C for 3 minutes in order to erase the thermal history. Cooling was conducted at a rate of 50 °C/min to -30 °C to keep the amorphous part, so as to make the glass transition temperature (T_g) more obvious. The second heating trace was from -30 °C to 200 °C at a heating rate of 10 °C/min, and the second cooling rate was the same. The heating and crystallisation thermogram was also measured. The temperature of the peak and area of the exothermic curve were taken as the crystallisation temperature (T_c). As soon as the temperature reached -40 °C, it was re-heated again at a rate of 10 °C/min, and the melting thermogram was measured. The glass transition temperature (T_g), and temperature of the peak and area of the endothermic curve were taken

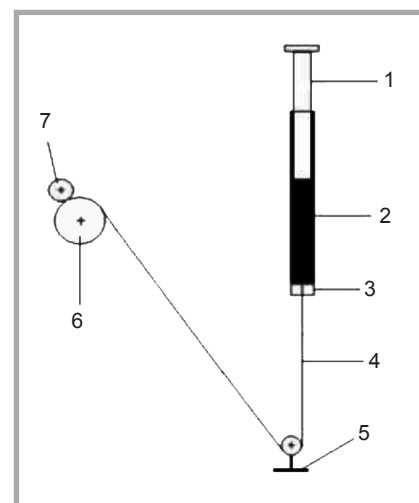


Figure 1. Schematic illustration of haul-off drafting device: (1) pressure lever, (2) capillary tube (polymer melt), (3) die, (4) melt, (5) roller, (6) program controlled drafting disk, (7) collection disk.

as the melting temperature (T_m) and heat of fusion (ΔH_f), respectively.

X-ray diffraction

An X-ray diffraction (XRD), ARL X'TRA X-ray powder diffractometer (Thermo Electron Corp, USA) was used with a diffractometer equipped with Cu Ka radiation. Film samples were prepared by hot-pressing using an iPP/LMPP composite masterbatch. The intensities of XRD were recorded from $2\theta = 10$ -50° with continuous scanning, a step size of 0.02° and scanning rate of 3 °/min. The degree of crystallinity of the iPP, LMPP and iPP/LMPP blends was calculated using Jade 5.0 software.

As-spun fibre preparation

A rheometer accessory (Haul-off drafting device) (Rosand RH7, UK) was used to prepare iPP/LMPP as-spun fibres at room temperature (25 °C). As in practical application, the spinning temperature was set at 190 °C, and the drawing and collecting roller speed was 30 m/min. Using this device, iPP/LMPP monofilaments were prepared with a linear density about 0.7 dtex. A schematic illustration of the device and its parts are shown in **Figure 1**.

iPP / LMPP drawn fibre mechanical properties

Using an INSTRON 2365 universal testing machine (USA), the mechanical properties of iPP/LMPP drawn fibres were investigated. The testing conditions for iPP/LMPP fibres were 25 ± 2 °C tem-

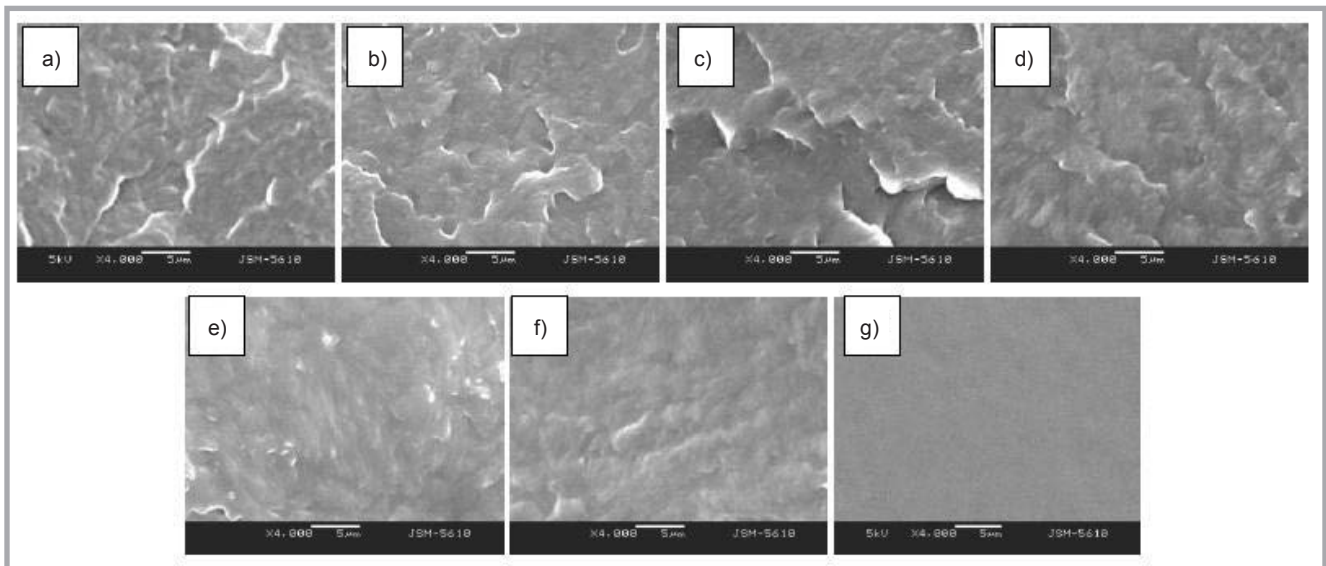


Figure 2. SEM images of cryo-fractured surfaces of iPP/LMPP blends: a) iPP, b) 5%, c) 10%, d) 15%, e) 20%, f) 25% and g) LMPP.

perature and 62-68% relative humidity. Prior to the experiment, the first upper and lower gripper distance was set at 5 cm apart, and then 10 cm fibres were placed between the upper and lower clampers in the intermediate position. The linear density of iPP/LMPP monofilaments was about 0.7 dtex.

Results and discussion

Morphology of iPP/LMPP blends

SEM micrographs of iPP/LMPP blends with varying contents are shown in **Figure 2.a-g**. Variations in the surface structures are apparent. SEM images of pure iPP and LMPP are totally opposite in morphology; the iPP surface is rough and not smooth as compared to the LMPP's. With

the images of iPP/LMPP blends, the phase interface is not distinguished due to the good miscibility between iPP and LMPP. As the amount of LMPP increases in the blends, their surface becomes smoother and more compact in structure. From **Figure 2.f**, it can be seen that 25% of the blended surface is the smoothest among the other blends and close to the LMPP surface morphology.

Dynamic mechanical analysis

For the glass transition (T_g) of all the samples, blends were heated at -30 to 100 °C temperature in a glass transition analyser (DMA Q800). The T_g of the LMPP was found to be 13.5 °C, which is higher than for the iPP and iPP/LMPP blends. The transition peak and peak area

of the LMPP are the most obvious and the highest, which shows the amorphous phase of LMPP. From **Figure 3**, we can observe that the T_g of iPP is at 8.0 °C, showing a small transition peak and area. It can be also be seen in **Figure 3** that the T_g of iPP/LMPP blends increases as the LMPP content increases in the blends; however, all the T_g values of the LMPP blend ratio are between pure those of iPP and LMPP, which indicates the good miscibility of iPP and LMPP.

Thermal behaviour of iPP/LMPP blends

Results of the thermal behaviour of iPP/LMPP blends are shown in **Figure 4**. In DSC heating curves, the temperature domain of glass transition T_g of pure LMPP was obviously found at -10.2 °C (**Figure 4.a**), while for the pure iPP and iPP/LMPP blends this was not found. This transitional change is due to the high crystallisation rate and high crystallinity of iPP and its blends, with the LMPP turning out to have a scarce amorphous region; thus the T_g signal is very weak. On the contrary, the T_g of LMPP is obvious because of its low crystallization rate and low crystallinity, which was verified by its cold-crystallisation peak at about 24.5 °C. The phenomenon of the LMPP re-crystallization process occurs when the molecular chain gets more dynamic at relatively high temperatures. However, an obvious cold-crystallization peak in the pure iPP and iPP/LMPP blends was not found.

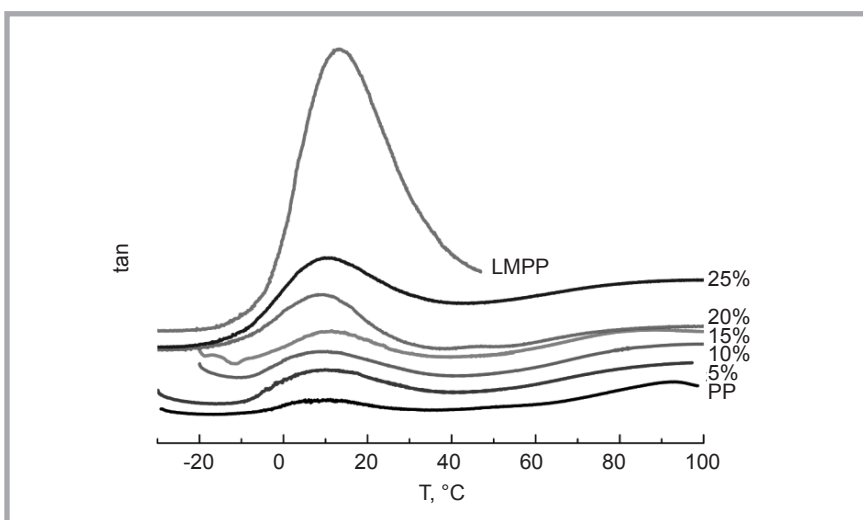


Figure 3. Glass transition temperature of iPP, LMPP and iPP/LMPP blends from -30 °C to 100 °C.

Compared with the melting peaks of the LMPP, iPP, and iPP/LMPP blends, that of

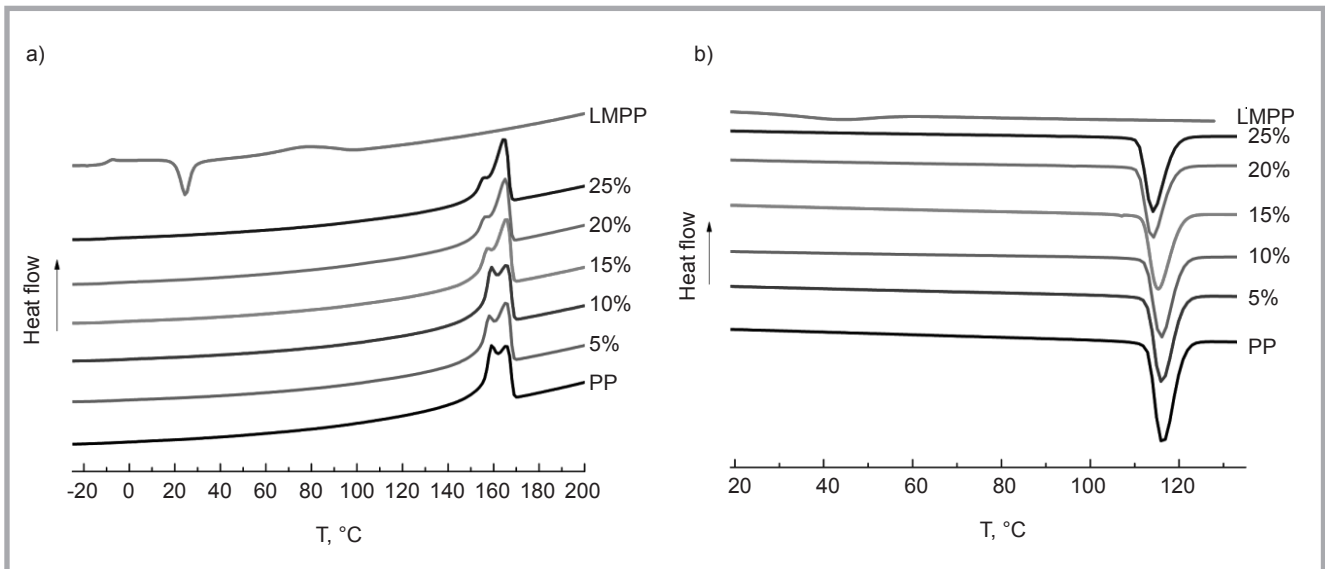


Figure 4. Influence of LMPP contents on a) second-heating curves and b) cooling curves of iPP/LMPP blends.

LMPP is low and wide, which indicates the low crystallinity of LMPP. The melting temperature of LMPP is 79.1 °C and that of iPP is double as compared to LMPP (**Figure 4.a**). There are some similarities and differences between the melting peaks of iPP and the blends with different LMPP contents. They all showed double melting peaks due to the defective crystalline being subjected to the melting re-crystallisation process, with the second melting peak temperature being all same, at about 165.5 °C (**Figure 4.a**). The first melting peak temperature and peak height both gradually shift to being lower with increased LMPP content, which suggests LMPP of relatively low molecular weight can act as diluent on iPP, with the defective part of crystalline becoming less. A small amount of LMPP (5% to 10%) may improve the crystalline perfection and crystalline uniformity of iPP. **Figure 4.b** shows cooling traces of LMPP, pure iPP and iPP/LMPP blends, with the lower and wider range of crystallization temperature indicating the crystallisation rate and low crystallization capacity of LMPP. Details of the thermal properties of iPP, LMPP and iPP/LMPP can be seen in **Table 1**.

X-ray diffraction of iPP/LMPP blends

Figure 5 shows the XRD intensity of the LMPP, iPP and iPP/LMPP blends. The iPP shows typical α -form peaks at 2θ angles of 14.0°, 16.8°, 18.5°, 21.1° and 21.7°, according to the Miller index of (110), (040), (120), (131) and (041) [19]. While the LMPP shows a more amorphous part with a low reflective peak.

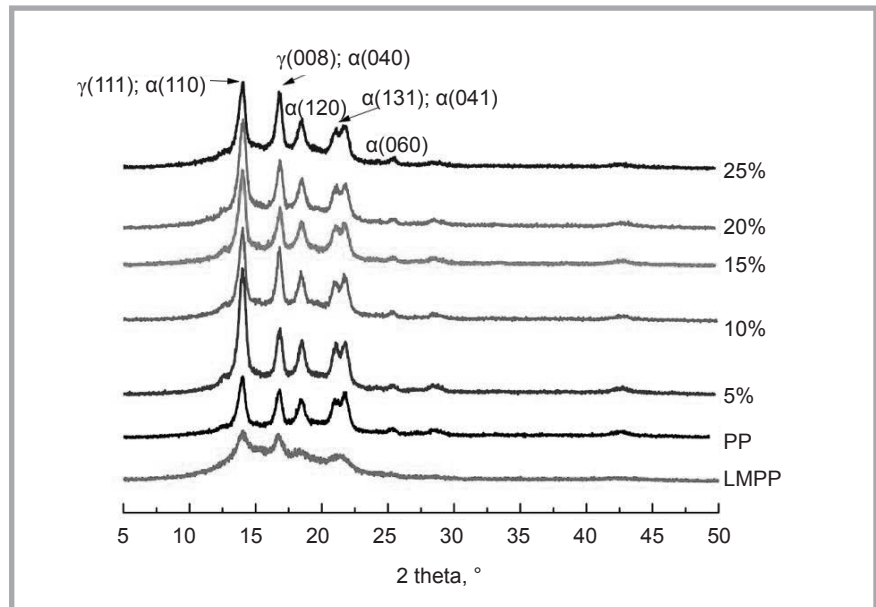


Figure 5. XRD intensity pattern of LMPP and iPP blends of isothermal crystallisation at room temperature.

Table 1. Thermal properties of iPP/LMPP blend samples.

iPP/ LMPP	T_{c1} , °C	T_{M1} , °C	T_{M2} , °C	ΔH_f , J/g
100/0	116.3	159.1	165.5	91.0
0/100	40.1	79.1	–	20.6
95/5	116.0	158.2	165.5	86.5
90/10	115.8	159.1	165.5	81.9
85/15	115.3	157.4	165.5	77.4
80/20	114.2	156.5	165.1	72.8
75/25	114.0	155.3	164.7	68.3

Table 2. Values of crystallinity of iPP/LMPP blends calculated using XRD.

iPP/LMPP	100/0	0/100	95/5	90/10	85/15	80/20	75/25
Crystallinity, %	40.2%	16.5%	43.2%	43.9%	42.5%	38.8%	38.3%

Table 3. Average mechanical properties of iPP/LMPP fibres under low drawing speed.

Sample	Tensile strength, MPa	Elongation at break, %	Elastic modulus, MPa
PP	52.8±1.2	2.20±0.12	753.7
5% LMPP	53.1±2.1	9.00±0.35	735.2
10% LMPP	59.3±3.3	10.36±0.36	613.5
15% LMPP	50.6±1.3	10.76±0.41	599.7
20% LMPP	52.1±1.8	11.62±0.53	563.4
25% LMPP	55.6±2.3	12.95±0.83	447.3
LMPP	25.3±0.5	9.50±0.91	89.4

Characteristic peaks of the presence of γ -form of iPP usually can be found at 2θ angles of 13.84°, 15.05°, 16.72°, 20.07°, 21.2° and 21.88°, corresponding to (111), (113), (008), (117), (202), and (026) [20]. Therefore there may be the co-existence of the α -form and γ -form in the first two peaks. With more content of LMPP blended with iPP, there is no obvious difference among the 2θ angles, which suggests that the crystal type does not change. However, the intensity ratio between the first two reflection peaks changes, which is due to the change in crystallinity and size of the crystals.

From **Table 2**, it is worth noting that the degree of crystallinity of iPP/LMPP blends increases and then decreases. With the increasing of LMPP content, the degree of crystallinity of iPP blended with 5-15% of LMPP is higher than that of pure iPP, while for iPP blended with 20-25% LMPP, it is lower. A similar phenomenon of change in the degree of crystallinity with iPP blends can be found elsewhere [19].

The blends with lower LMPP contents, for instance iPP/LMPP (5% to 15%), the degree of crystallinity increased, compared to pure iPP, while the degree of crystallinity of 20% to 25% blends decreased. A possible reason for expanding crystallinity is suggested by the diluent LMPP molecular suppression entanglement between iPP molecules and the mobility of iPP molecules during crystallization. For higher content blends of LMPP, the decreasing crystallinity of iPP blends may due to a higher amount of diluent LMPP activity and LMPP molecular restrains, diffusing to the surface during the crystallization. It was also found that LMPP is a low crystalline polymer, which when blended with iPP homopolymer can act as a diluent. Numerical crystallinity values of the iPP, LMPP, and iPP/LMPP blends can be seen in **Table 3**, which were calculated with the help of Jade 5.0 software.

Mechanical properties of iPP/LMPP blends fibres

The tensile strength of the iPP/LMPP blends fibres is shown in **Table 3**. It can be seen that the elongation at break of pure PP fibre is the lowest, while the elastic modulus is the highest. The elongation at break increased with the increasing of LMPP content, with the maximum elongation at break of the LMPP 25% blend reaching 12.95%, which shows potent stretchability. The elastic modulus of iPP/LMPP blends also decrease with the adding of LMPP, which indicates the fibre's smoothness and softness. This phenomenon occurred due to the outstanding stretchability and low modulus of LMPP. The tensile strength of pure iPP fibre was lower as compared to the 5% and 10%. On the other hand, the tensile strength of the 10% blend fibres was found to be strongest, whose peak value was near 60 MPa. Moreover XRD crystallinity data of the 10% blend was the highest, which can be seen in **Table 3**. As the LMPP was content increased to more than 25%, the strength of the blended fibres decreased, which suggests the low strength of pure LMPP.

Conclusions

- Blends of iPP and LMPP were made with varied blend ratios. As the LMPP content increased in the blends, miscibility was improved and the surface became smoother.
- DSC results showed an increase in the melting-point of pure LMPP from 79.1 °C to 165.5 °C in all iPP/LMPP blends tested.
- The blends with LMPP contents higher than 15% showed a decrease in the degree of crystallinity compared to pure iPP. The glass transition temperature of pure iPP was found to be 8.0 °C, while and LMPP was at 13.5 °C.
- The elongation at break increased with an increase in the LMPP amount, with the maximum elongation at break of the LMPP 25% blend being able to

reach 12.95%, which shows excellent stretchability. The elastic modulus of iPP/LMPP blends also decreases with the addition of LMPP, which indicate the fibre's softness. On the other hand, the elongation at break of pure PP fibre is the lowest, but the elastic modulus is the highest.

- The results from this study suggest that suitable LMPP contents with iPP could be useful in producing smoother non-woven fabrics.

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- antibacterial activity of textiles **PN-EN ISO 20743:20013**
- method of estimating the action of microfungi **PN-EN 14119:2005 B2**
- determination of antibacterial activity of fibers and textiles **PN-EN ISO 20645:2006**.
- method for estimating the action of microfungi on military equipment **NO-06-A107:2005** pkt. 4.14 i 5.17

Tests not included in the accreditation:

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