PREPARATION AND CHARACTERIZATION OF RICE STARCH AND TRANSITION METAL STEARATES FILLED POLYPROPYLENE

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Master of Science

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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LIST OF SYMBOLS

T_c	Crystallization temperature
T_m	Melting temperature
T_g	Glass transition temperature
rpm	Rotation per minute
w_f	Final weight of sample
w_i	Initial weight of sample
σ_t	Tensile strength (MPa)
F_{max}	Maximum load (N)
A	Cross sectional area of sample
L	Length at breaking point of sample
L_o	Initial length of sample
w_d	Final weight of sample
wt %	weight percentage
T_{max}	Maximum temperature at decomposition of sample

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
AWM	Accelerated Weathering Machine
AWT	Accelerated Weathering Test
$CoSt_3$	Cobalt Stearate
DSC	Differential Scanning Calorimetry
EB	Percentage of Elongation at Break
ED	Enzymatic Degradation
$FeSt_3$	Iron Stearate
FTIR	Fourier Transforms Infrared Spectroscopy
MAPP	Maleic Anhydride Polypropylene
PCL	Poly (∈ - Caprolactone)
PE	Polyethylene
WT %	Per Hundred Resin
PLA	Poly Lactic Acid
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
RS	Rice Starch
SEM	Scanning Electron Microscopy
TGA	Thermo Gravimetric Analysis
TM	Tensile Modulus
TMS	Transition Metal Stearates
TRS	Treated Rice Starch
TS	Tensile Strength
URS	Untreated Rice Starch
UV	Ultra Violet
WA	Water Absorption

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ABSTRAK

Fleksibiliti tinggi plastik sintetik dari segi sifat mekanikal dan ketahanan telah menjadi faktor kemerosotan persekitaran semula jadi. Kajian ini telah dijalankan ke arah pembangunan dan penggunaan plastik degradasi sebagai alternatif untuk menggantikan plastik komoditi. Kanji beras yang tidak dirawat (URS), kanji beras dirawat (TRS) dan stearat logam peralihan (TMS) digunakan sebagai pengisi dan digabungkan dengan polipropilena (PP). URS, TRS dan TMS diisi PP telah disediakan melalui teknik ekstrusi dan penyuntikan acuan. URS dan TRS dicampur dengan PP dengan kepekatan masing-masing 10, 20, 30, 40 dan 50 wt %. Komposisi TMS ($CoSt_3$ dan $FeSt_3$) sebanyak 0.1, 0.3, 0.5, 0.7 dan 0.9 phr digabung dengan PP. Campuran yang disediakan dianalisis dari segi sifat mekanikal seperti kekuatan tegangan (TS), modulus tegangan (TM) dan peratusan pemanjangan pada takat putus (EB). Ciri-ciri termo-oksidatif telah dikaji dengan menggunakan analisis termogravimetrik (TGA) dan kalori simetri pengimbasan (DSC). Kadar degradasi telah dikaji dengan menggunakan degradasi enzimatik dan ujian kaji cuaca yang dipercepatkan. Ciri-ciri degradasi dan morfologi permukaan diperhatikan menggunakan spektroskopi inframerah transformasi fourier (FTIR) dan analisis mikroskop elektron (SEM). Keputusan menunjukkan TS dan TM sampel yang mengandungi URS dan TRS berkurangan dengan peningkatan kandungan URS dan TRS. Keputusan menunjukkan TS dan TM maksimum kedua-dua $CoSt_3$ dan $FeSt_3$ pada kepekatan TMS sebanyak 0.5 phr. Analisis DSC menunjukkan bahawa entalpi dan peratus penkristalan sampel yang mengandungi URS dan TRS menurun. manakala peratus penkristalan sampel PPFe5 lebih tinggi berbanding sampel PPFe5. Tambahan pula, T_{max} yang lebih tinggi dalam sampel TRS diisi dengan PP berbanding dengan sampel URS diisi dengan PP membuktikan TRS meningkatkan kestabilan terma sampel. Keputusan TGA menunjukkan bahawa PPCo5 mempunyai T_{max} yang lebih tinggi berbanding dengan PPFe5. $CoSt_3$ yang mempamerkan suhu penguraian yang lebih tinggi berbanding $FeSt_3$. Melalui degradasi enzimatik, sampel yang mengandungi URS dan TRS mengalami pengurangan berat badan tertinggi selepas 10, 20 dan 30 hari untuk 50 wt % berat URS dan TRS. Pengurangan berat dalam kedua-dua sampel PPCo5 dan PPFe5 sangat sedikit selepas degradasi enzim. Perubahan warna yang ketara dapat diperhatikan dalam sampel yang mengandungi URS dan TRS selepas ujian kajicuaca yang dipercepatkan. Ujian sama menunjukkan bahawa keupayaan $CoSt_3$ sebagai pro-oksida yang lebih baik daripada $FeSt_3$ seperti yang dilihat dalam penampilan visual dan analisis SEM. Penggabungan URS, TRS dan TMS ke dalam matriks PP mempamerkan beberapa perubahan ketara ke atas sifat campuran plastik yang dihasilkan. Dalam semua campuran, penurunan sifat mekanikal dan terma telah diperhatikan tetapi peningkatan yang ketara dalam kadar degradasi sampel boleh diperhatikan. Penggantian matriks PP dengan pengisi dapat meningkatkan kecenderungannya ke arah degradasi. Dari analisis data, TMS bertindak sebagai pengisi terbaik di antara ketiga-tiga pengisi yang telah diadun dengan PP diikuti oleh TRS dan URS. Secara ringkasnya, campuran pengisi semula jadi dan pro-oxidan yang berasaskan plastik sintetik boleh menjadi penyelesaian yang baik untuk menyelesaikan isu-isu alam sekitar yang disebabkan oleh sisa plastik yang tidak terbiodegradasi.

ABSTRACT

The high versatility of synthetic plastics in terms of mechanical properties and durability has become a deteriorating factor of natural environment. In response to these issues, this study has been directed towards development and application of degradable plastics as an alternative to replace commodity non degradable plastics. Untreated rice starch (URS), treated rice starch (TRS) and transition metal stearates (TMS) were used as fillers and incorporated with polypropylene (PP). URS, TRS and TMS filled PP samples were prepared through extrusion and injection molding technique. URS and TRS were mixed with PP with a concentration of 10, 20, 30, 40 and 50 wt % respectively. Composition of TMS ($CoSt_3$ and $FeSt_3$) of 0.1, 0.3, 0.5, 0.7 and 0.9 phr was blended with PP. The prepared blends were characterized by tensile properties such as tensile strength (TS), tensile modulus (TM) and percentage of elongation at break (EB). The thermo-oxidative properties were studied by using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Degradability of blends was studied by using enzymatic degradation and accelerated weathering test. The degradation properties and surface morphologies were observed using fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM) analysis. The results indicated that TS and TM of URS and TRS filled PP samples reduced as the composition of starch increases. The results indicated that maximum TS and TM of both $CoSt_3$ and $FeSt_3$ at concentration of TMS of 0.5 phr. DSC analysis showed that enthalpy and crystallinity of URS and TRS filled PP samples dropped as the composition of URS and TRS increased from 10- 50 wt % meanwhile crystallinity of PPFe5 samples were much higher than PPCo5 samples. Furthermore, higher T_{max} in PP/TRS compared to PP/URS signified that the incorporation of TRS improved the thermal stability of the blends with the increase in the composition of starch (10-50 wt %). TGA results showed that PPCo5 has a higher T_{max} as compared to PPFe5 and $CoSt_3$ exhibits higher decomposition temperature as compared to $FeSt_3$. During enzymatic degradation, URS and TRS filled PP samples experienced highest weight reduction after the 10, 20 and 30 days for 50 wt % URS and TRS. The weight loss in both PPCo5 and PPFe5 samples are almost negligible after enzymatic degradation. Significant discoloration was observed in both URS and TRS filled PP samples after accelerated weathering test. Same test showed that the ability of $CoSt_3$ as pro-oxidant is more deteriorating than $FeSt_3$ as observed in visual observation and SEM analysis of samples. The incorporation of URS, TRS and TMS into PP matrices exhibits some significant changes on the properties of plastic blends been produced. In all the formulation, it was observed decrease in mechanical and thermal properties but a significant improvement in the degradability of samples can be observed. Substitution of PP matrix with fillers can improve its susceptibility towards degradation. From the data analysis, TMS acts as best filler among all the three fillers been blended with PP followed by TRS and URS. Conclusively, filling of natural filler and pro-oxidants into synthetic plastics can be a great solution to solve the environmental issues caused by non-degradable plastic wastes.

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