



Utilization of Acrylic Modified PET and Its Nanocomposite Fibers in Transdermal Medical Applications

Shaymaa E. Elshafey ¹, Wael S. Mohamed ^{*2} and Rokaya A. Sobh ²

¹ Physical Chemistry Department– National Research Centre

² Polymers & Pigments Department– National Research Centre



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In Loving Memory of Late Professor Doctor "Mohamed Refaat Hussein Mahran"

Abstract

Polyethylene terephthalate /Montmorillonite clay nanocomposite was prepared using commercially modification Montmorillonite (MMT). The prepared nanocomposite was characterized using scanning electron microscope (SEM). Fibers of pure Polyethylene terephthalate (PET) and Polyethylene terephthalate /montmorillonite clay nanocomposite (PET/MMT) were produced using melt spinning technique. Grafting of Acrylic acid was performed using grafting emulsion polymerization technique to obtain PET/MMT-g-AA fibre and the prepared fibers were characterized using mechanical properties , FTIR in addition to SEM analysis. The results show that the PET/MMT-g-AA fibre have higher mechanical properties, and lower water absorption than pure PET and PET/MMT fibers in addition to high degree in homogeneity between PET and MMT clay in PET/MMT fibre. Clindamycin drug was successfully loaded into PET, PET/MMT and PET/MMT-g-AA fiber via solvent immersion method with different drug fiber ratio in presence of glyoxal crosses linker. Drug release profile was studied for 120 min and the results showed that about 41% from the initial loaded drug was released from the fiber after an hour of release starting time.

Key Words: Polypropylene / tallow /Montmorillonite / Melt Spinning / clindamycin / transdermal / drug delivery

1. Introduction

PET/MMT melt spun fibers are created by combining PET polymer with montmorillonite clay nanoparticles through a spinning process, resulting in hybrid fibers that have advantageous properties from both PET and MMT. PET is a widely used synthetic polymer known for its mechanical properties and chemical resistance, making it ideal for drug delivery systems. MMT, on the other hand, is a natural clay mineral known for enhancing properties like mechanical strength and thermal stability. These PET/MMT fibers have gained attention for their potential in transdermal drug delivery, offering controlled and sustained release of therapeutic agents (1-4).

To produce PET/MMT fibers, the melt spinning process is commonly used. This involves melting the polymer and extruding it through fine nozzles into an air-filled chamber, where it solidifies into continuous fibers. Adding MMT during this process improves fiber quality and enhances drug release properties. Transdermal drug delivery is a non-invasive method that allows drugs to be absorbed through the skin, offering advantages like prolonged drug release and reduced side effects. PET/MMT fibers play a crucial role in transdermal drug delivery by providing

controlled and sustained release of therapeutic agents (5-8).

PET/MMT fibers overcome limitations associated with conventional drug delivery systems. Transdermal delivery avoids metabolism by the liver, increasing drug bioavailability. Additionally, the controlled release offered by these fibers improves patient compliance and reduces potential side effects (9-10). Studies have shown the potential of PET/MMT fibers in delivering various drugs, such as anti-inflammatory agents, analgesics, antibiotics, hormones, and anticancer drugs. These fibers have demonstrated sustained drug release, biocompatibility, and controlled release kinetics. In transdermal drug delivery, grafting acrylic acid monomer into the melt spun polypropylene montmorillonite nanocomposite fabric offers advantages like improved drug permeability, sustained release properties, enhanced barrier properties, and increased drug-loading capacity (11-12). Clindamycin, an antibiotic used to treat bacterial infections, can be delivered topically through gels or creams using PET/MMT fibers. Transdermal delivery of Clindamycin bypasses systemic side effects and delivers the drug directly to the infected area, providing a sustained therapeutic

*Corresponding author e-mail: wsabry1976@yahoo.com , (Wael S.Mohamed).

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effect. This method is particularly beneficial for acne treatment, as it can effectively kill acne-causing bacteria in hair follicles and on the skin surface (13-14).

Acrylic modified PET and its nanocomposite fibers hold significant significance and relevance in transdermal medical applications in the field of chemistry. Their favorable properties, including biocompatibility, durability, and the ability to incorporate nanoparticles, make them ideal candidates for developing advanced transdermal drug delivery systems. Their use in transdermal medicine can revolutionize drug delivery, improving patient outcomes and quality of life.

The aim of this research is to prepare PET/MMT melt spinning fibre and modify the surface of the produced fibre by grafting of acrylic polymers in order to compare the properties of the grafted PET/MMT melt spinning fibre with the unmodified fibre to determine the effectiveness of the grafting process on enhancing the surface properties and using the modified surface fibre in transdermal drug delivery of Clindamycin drug where Grafting acrylic polymers onto the surface of PET/MMT melt spinning fibre will enhance its surface properties, such as hydrophobicity or hydrophilicity, which can make it suitable for specific applications..

2. Experimental

2.1. Materials

Modified MMT-682 608 (25-30 wt % trimethylstearyl ammonium) was supplied by Sigma-Aldrich – Germany.

PET flakes (RT20) were provided by the company Invista Resins & Fibers. The PET-RT20 is characterized by an intrinsic viscosity (IV) of 0.634 in dichloroacetic acid, a flake size of 4 mm and a bulk density of 0.9 g·cm⁻³.

Glyoxal was purchased from Nen Tech Ltd. Brixworth Northants. U.K. Potassium dihydrogen phosphate (KH₂PO₄) was purchased from GenLab (packaged in Egypt). Ethanol and sodium hydroxide were purchased from El-Nasr Company - Egypt. Clindamycin drug was provided as a gift drugs from Smith Kline Glaxowellcome Company, Egypt.

2.2. Preparation of PET / MMT masterbatche

To avoid degradation of the polymers during extrusion, the nanoclay and polymer were dried and stored in air tight vessels until usage.

For compounding the MMT powders and polymer granulate were premixed in quantities of 1 wt. % MMT content. The mixtures were extruded as polymer strands using a HAAKE twin screw extruder, cooled in a water bath and pelletized. The granulate was dried and stored in air tight vessels until further

usage.

2.3. Multifilament spinning

Spinning of the fibers was done using a HAAKE single screw extruder using a spinneret with 32 holes of 300 μm diameter. One filter layer with 90 μm was used. A spinning pump with 1.2 ccm was used with 30 rpm. Dryfi L 165 M was used as spin finish.

2.4. PET / MMT Fibre Production via Melt Spinning Method (12)

Before the fibre production, the PET / montmorillonite nanocomposite was dried at 0.1 bar and 160 oC. Continuous fibres were produced via spinning process using extruder Haake 9000 (Germany). The solid polymeric pellets were introduced in the spinning machine which has three independent temperature areas and is equipped with a pressure sensor. In order to process the PP/MMT clay nanocomposite filament, the heating temperature is regulated between 180 and 230 oC. As a spinning pump a toothed wheel pump (or gear pump) is available, whose delivery volume is 0.6 cm³. The control of the temperature at the pump and the spinneret is affected by a thermal element where a spinning nozzle a 10-hole-spinneret (diameter of pore 200 μm) is used after turning the spinning-fiber around two galettes, it is spooled on a coil by a coiler (winder) from Barmag, Type SW 66 SSD.

2.5. Grafting emulsion polymerization of acrylic acid onto polypropylene nanocomposite fibres (15)

1g in weight of PET/MMT melt spun nanofibre were placed separately in a stoppered glass vessel containing 20 mL distilled water, Potassium persulfate (PPS) as an initiator (1x10⁻³ mol/l), and 1% sodium lauryl ether sulfate (SLES) as an emulsifier, then the vessel was gently shaken for 10 minutes with a thermostatic shaker water bath (HWT- 10C with temperature range up to 100°C and speed up 200 rpm, China). 5% Acrylic Acid (AA) monomer was added to the mixture. Then the temperature was adjusted to 60°C and the graft polymerization was allowed to continue under shaking for the period specified (4 hr). After that, the homopolymer and unreacted components were removed from the modified melt spun fiber by washing it with distilled water. The obtained grafted copolymer PET/MMT-g-AA nanofibre were then dried at 40°C for 12h and the grafting yield percentage has been calculated using the below Equation:

$$\text{Grafting yield (\%)} = [(W_g - W_o) / W_o] \times 100$$

Whereas W_g = weight of the fibre after grafting, W_o = weight of the fabric before grafting.

Clindamycin drug loading into Acrylic acid grafted PET / MMT nanocomposite fibres using solvent

immersion method[16].

Clindamycin drug (Figure 1) loading was done using a solvent immersion method by dissolving clindamycin drug in ethanol (200mg/ml) then; different amount of PET/MMT-g-AA nanocomposite fibre samples added to the solution in presence of glyoxal cross linker in absence of light. The mixture was brought to equilibrium overnight under gentle stirring in closed container using magnetic stirrer then, filtered by vacuum filtration using a Büchner funnel with the filter flask attached to a water aspirator.

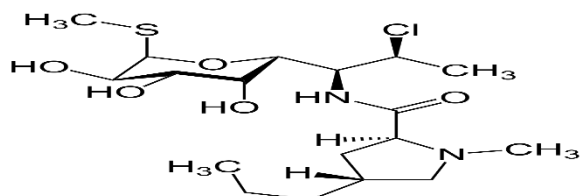


Figure1: clindamycin drug chemical structure

2.6. Drug entrapment efficiency (EE%)

To determine the drug entrapment efficiency (EE %) in loaded onto the fiber was determined by an indirect method, through measuring the free drug (unloaded drug). The initial drug concentration and the drug concentration in the solution after finishing the immersion process was determined by measuring the absorbance at 263 nm on a Shimadzu Ultraviolet-visible spectrophotometer with double beam using a standard calibration curve experimentally obtained with methanol solutions.

The drug EE was defined as the ratio of the weight of the drug loaded into the spinning fiber to the weight of the drug initially used (17).

$$E.E. (\%) = \frac{\text{The weight of the drug loaded into the spinning fiber}}{\text{The weight of the drug initially used}} \times 100$$

2.7. In-Vitro release of clindamycin [18]

In this study, the effects of the ratio of the drug to fiber carriers on the in vitro release of clindamycin (CDM), loaded fiber was evaluated.

After the solvent immersion process, the drug loaded carrier fibres were collected using a rotary evaporator. In comparison with the other drying methods, the utilization of rotary evaporation in the loading process has shown significant advantages, such as process simplification by avoiding filtration to reduce the risk of crystallization of the drug on the surface of the carriers, fixing amounts of the loaded drug and providing industrial scalability for loading the drug into the fibers.

The clindamycin drug releases from the drug-loaded fibres at different loading conditions were performed in a phosphate buffered saline media of pH 7.4 using a dialysis bag technique. Buffer solution of

pH 7.4 was prepared by mixing 250 ml of 0.1M KH₂PO₄ and 195.5 ml of 0.1M NaOH. Dialysis sacs were equilibrated with the dissolution medium for few hours before experiments.

2.8. Characterization

The morphology and fracture surface of the polymer and its composite were examined by scanning electron microscope (SEM) analysis using a Zeiss DSM 962 microscope.

The amount of free drug and drug released were determined by using the ultraviolet-visible recording spectrophotometer (Cary 500, Varian) equipped with a quartz cell having a path length of 1 cm at λ -max 210 nm.

The mechanical properties of the PET/MMT fibers were measured with a Zwick Zugprüfgerät machine (Ulm, Germany) with a force of 10 KN. (ASTM D-3822-07)

3. Results & Discussions

3.1. Preparation of Polyethylene terephthalate / Montmorillonite Nanocomposite

3.1.1. Scanning Electron Microscope

The morphology investigation of pure PET and PET/ modified MMT nanocomposite prepared by direct melt intercalation method is achieved by scanning electron microscope (SEM) (X500) and is present in figure 2.

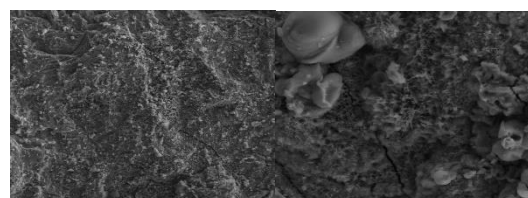


Figure 2: A) Pure PET and B) PET/MMT nanocomposite

A uniformly homogenous dispersion of the modified clay without agglomeration is observed in the PET matrix which is considered an indication of the successful preparation of the PET/MMT nanocomposite.

3.2. Polypropylene / Montmorillonite Fibre Production , Grafting of Acrylic acid monomer onto PET/MMT fibre and loading of Clindamycin drug

MMT incorporation into the PET fibre through PET melt spinning is mainly carried out for enhancement the PET fibre properties

Grafting of 5% Acrylic acid onto PET /MMT nanocomposite fibre was carried out using emulsion grafting polymerization technique to produce PET/MMT-g-AA. The grafting percentage was

calculated and reached 87% after 4 hours.

3.2.1. Clindamycin drug loading

A certain weight of PET, PET/MMT and PET/MMT-g-AA spinning fibre are immersed in Clindamycin drug in ethanol solution (200mg/ml) in presence of 5% glyoxal for 60 min under shaking. Effect of drug / fibre concentration ratio and time of fibre immersion was studied (19).

3.2.1.1. Effect of drug /fibre concentration:

Three different drug / fibre concentration ratio which are 1:1, 1:2 and 1:3 was studied and the drug entrapment efficiency was determined for each ratio by determining the drug concentration in the solution after finishing the fibre immersion process.

Entrapment efficiency (E.E) % was calculated for each ratio and the data are recorded in table 2. From the recorded data, it was concluded that, E.E % is inversely proportional with the fibre ratio in both spinning fibres used. From the data, it is also clear that E.E % for the PET/MMT-g-AA nanocomposite fibre is higher than that of the pure PET fibre due to presence of MMT clay which increases the available active sites for drug adsorption (20-21).

The high surface area of MMT and increasing Van der Waals forces led to enhancement of drug adsorption in case of PP/MMT spinning fibre.

Table 1: Entrapment efficiency % of Clindamycin drug loading into PET and PET/MMT spinning fibres using different drug/fibre ratios.

Drug/fiber ratio	EE %		
	Pure PET fibre	PET/ MMT fibre	PET/ MMT-g-AA fibre
1:1	38.5	56.8	69.5
1:2	30.6	48.5	66.4
1:3	29.6	41.5	61.5

3.2.1.2. Effect of time of fibre immersion:

Clindamycin drug / fibre ratio 1:1 using PET/MMT-g-AA spinning fibre was immersed in ethanol solution in presence of glyoxal cross linker for different time intervals and the E.E % was calculated for each time interval. The result data listed is table 3. From the data, it can be concluded that, the drug adsorption is very rapidly at first interval and reached maximum after 60 min then it remains constant.

3.2.2. Clindamycin drug release profile

Figure 3 represents the Clindamycin release profile from drug loaded PET/MMT-g-AA spinning fibre with fibre/drug ratio 1:1 after 60 min drug loading time. During 120 min releasing time, rapid initial drug

release was observed for the first 30 min and reached to about 23% from the initial loaded drug, then the release rate decreased in the next 30 min till reached to plateau region after an hour of release starting time where the total release reached to about 41% from the initial loading drug.

Table 2: Entrapment efficiency (EE) % of Clindamycin drug loaded on PET/MMT-g-AA spinning fibre using drug/fibre ratio 1:1 in different time of fibre immersion.

E.E %	Time of fibre immersion					
	15 min	30 min	45 min	60 min	90min	120 min
	29.2	33.5	48.5	69.5	69.7	69.5

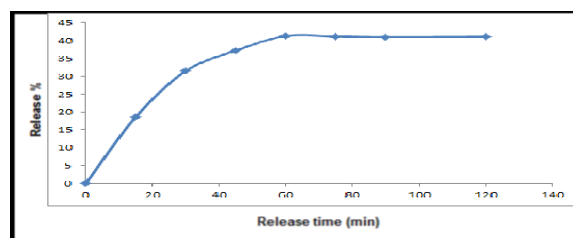


Figure 3: Clindamycin release profile from drug loaded PET/MMT-g-AA spinning fiber with fiber/drug ratio 1:1 after 60 min drug loading time.

3.2.3. Mechanical Properties

The mechanical properties were investigated for both pure PET and PET/MMT nanocomposite fibre in order to elucidate the MMT addition effect on mechanical properties of polypropylene fibre including tensile and elongation parameters (T.S, E and E*). The data obtained from mechanical properties investigation listed in table 1. This data refers to an increment in the fibre mechanical properties as a result of MMT addition which indicate the dispersion of MMT in the PET fibre.

3.2.4. Water Absorption

Water absorption for pure PET, PET/MMT and PET/MMT g- AA fibres was calculated through different time intervals. The data obtained from water resistance calculation are listed in table 2. Where the results indicate that, pure PET/MMT-g-AA fibre has to be less resistance to water absorption in comparison with both pure PET and PET/MMT fibre which indicates that both MMT clay and grafted Acrylic acid are useful for enhancement the PET fibre water repellence.

Grafting of Acrylic acid enhance both mechanical properties and water absorption of the prepared PET/MMT nanocomposite fibres due to blocking of the fibre porous by acrylic acid polymer.

Table 3: Mechanical properties and water absorption of pure PET and PET/MMT nanocomposite fibre

Sample	Mechanical properties			Water absorption (%)		
	Tensile Strength (T.S)	Elongation at break (E)	Elongation at Fmax (E*)	30 min.	60 min.	120 min.
Pure PET fibre	180.2	34.7	36.3	45.6	49.5	52.3
PET/MMT fibre	196.3	35.1	36.6	39.5	42.6	45.6
PET/MMT-g-AA fibre	220.0	38.2	38.0	25.4	28.4	30.0

3.2.5. Scanning Electron Microscope (SEM)

Morphology investigation for both pure PET, PET/MMT and PET/MMT-g-AA fibre was illustrated by SEM analysis and represented in figure 4.

The SEM analysis shows the smooth of pure PET fibre represents the high degree in homogeneity between the PET fibre and MMT clay which indicates the successfully preparation of PET / MMT nanocomposite. From the figure, it is clear that the grafting process was confirmed by presence of polymer coating morphology.

Loading of clindamycin drug onto PET/MMT-g-AA surface was confirmed by SEM image where it is clear that, the drug loaded fibre has a smooth texture.(23)

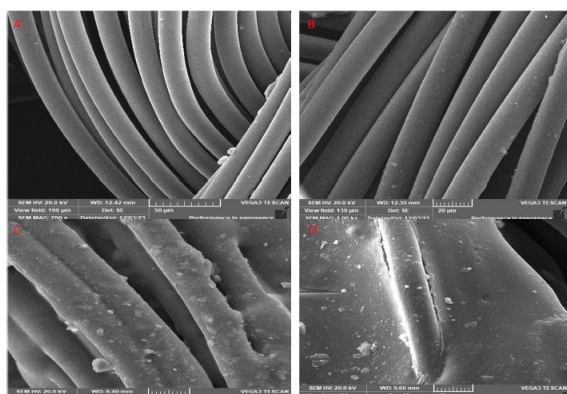


Figure 4: SEM of A) pure PET, B) PET/MMT, C)PET/MMT-g-AA and D) PET/MMT-g-AA loaded with Clindamycin drug fibres

3.2.6. FTIR/ATR analysis

Figure 5 represents FTIR of PET/MMT, PET/MMT-g-AA and PET/MMT-g-AA loaded with Clindamycin drug. The figure showed the appearance of Si-O group characteristic absorption band at 1092 cm⁻¹ PET/MMT nanocomposite patterns indicates the successfully preparation of PET / MMT nanocomposite fibre.

Grafting of Acrylic acid was confirmed by appearance of carbonyl group specific pattern at 1650 cm⁻¹ in the PET/MMT-g-AA.

Loading of Clindamycin drug was confirmed by ATR analysis by presence of N-H characterized peak at 1590 cm⁻¹ (bending) and 3400 cm⁻¹ (stretching) (24).

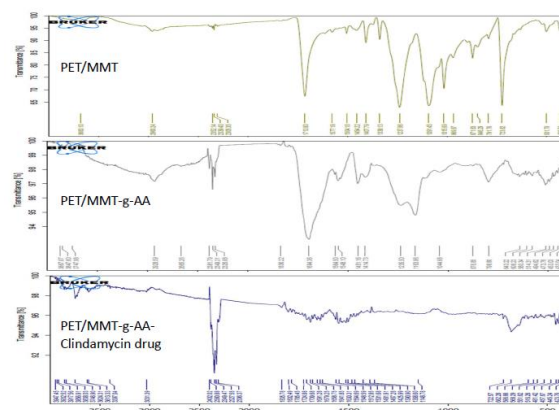


Figure 5: FTIR/ATR of A) PET/MMT, B)PET/MMT-g-AA and C) PET/MMT-g-AA loaded with Clindamycin drug fibres

4. Conclusion

Polyethylene terephthalate (PET) / Montmorillonite (MMT) nanocomposite was prepared using commercially modification of MMT clay

The prepared nanocomposite was characterized using SEM analysis a uniformly homogenous dispersion of the modified clay without agglomeration which is observed in the PET matrix confirms the successful preparation of the PET/MMT nanocomposite.

Pure PET and PET/MMT nanocomposite fibers were produced using melt spinning technique.

PET/MMT-g-AA fibre was obtained by grafting of 5% acrylic acid and the grafting percentage reached 87%. The prepared fibers were characterized using mechanical properties, water absorption in addition to SEM and ATR analysis which confirm the benefits of MMT and grafted Acrylic acid addition in the enhancement of PET fiber properties.

Clindamycin drug was successfully loaded into the PET/MMT-g-AA fibers via a solvent immersion method with different drug / fiber ratio in presence of glyoxal cross linker and the results showed that, E.E % for the PP/MMT nanocomposite fibre is higher than that of the pure PET fibre due to presence of MMT clay and reached to 56.8% for drug/fiber ratio 1:1 during an hour immersion time.

Clindamycin release profile from drug loaded PET, PET/MMT and PET/MMT-g-AA spinning fiber with fiber/drug ratio 1:1 after 60 min drug loading time was

studied and the results showed that the higher EE% reached about 41% from the initial loaded drug was released from PET/MMT-g-AA fiber after an hour of release starting time.

5. Acknowledgment

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6. Potential conflicts of interest

There are no potential conflicts of interest

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