

# **RECYCLING NEWSLETTER**

### **3RD QUARTER 2020**

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# **Committed to unlock the circularity of PS**



Alliance to End Plastic Waste: 2020 Progress Report

In This Issue: Upcoming SPE Recycling Division Events

Influence of Mechanical Recycling on Thermotropic LCP & GFR PP

## Editor's Corner

At the time of writing, we have just completed our first virtual event, part of the SPE "Plastics in..." series. "Innovations in Chemical & Mechanical Recycling – The Virtual Edition" was a 1-day event with speakers from Mars, UMass Lowell, and GreenBlue. With over 125 attendees, the event can be considered a great success for a maiden effort. Many of us know that plastics recycling is a critically important topic these days, and the level of attendance and discussion at a virtual event – despite a packed calendar – is testament to our Division's mission. For those who were unable to attend, a recording of all the sessions is available on the SPE website and on the Division microsite (https://recycling.4spe.org).

Upcoming events include two new webinars, free to SPE members, \$195 for non-members. Spread the word!

"This is Not the Wild West: Sustainability Definitions, Standards, and Regulations"<sup>°</sup> Tuesday, October 13, 11am EST Kelvin Okamoto, Green Bottom Line Consulting

*"Life Cycle Assessments: Why and How"* Wednesday, November 18, 11am EST Christoph Koffler, Sphera Consulting

In this edition of our newsletter, we offer a variety of content that covers both technical and practical developments in the world of plastics recycling. Our lead technical paper explores the effect of mechanical recycling on the properties of injection-molded PP reinforced with thermotropic liquid crystalline polymer (TLCP) or long glass fiber (GF) has been investigated. From the commercial world, we share a recent 'innovation brief' from Trinseo and Coexpan about the use of recycled polystyrene in form/ fill/seal technologies for dairy packaging. This is a large and important packaging market where PS has been under pressure from new developments in PP and PET. And from a global perspective, we are happy to share an abbreviated version of the Alliance to End Plastic Waste's (AEPW) annual summary.

Speaking of AEPW, SPE members are invited to participate in a new, global program sponsored by the Alliance: #ALL\_TOGETHER GLOBAL CLEANUP. In celebration of World Cleanup Day 2020, the goal of this global initiative is to help remove litter "one piece at a time" from local communities worldwide. Starting \*\*Saturday, September 19th\*\* and continuing through October 11th, ALL\_TOGETHER GLOBAL CLEANUP will unite thousands of volunteers from around the world to engage with their communities to discover, identify, collect and dispose of any litter they find.



The Alliance has joined with technology and innovation partner Litterati to provide an app that uses geo-tagged photos of litter to measure and track participation. For SPE members to participate, simply download Litterati from the App Store or Google Play Store, open the Litterati app and ENTER the code: 4SPE2020. Volunteers can then head out into their local community and use the camera in the Litterati app to take photos of the litter collected. SPE's challenge is to collect 1,000 pieces of litter!

One piece at a time, SPE members can show how individual impact is multiplied when we work ALL\_TOGETHER.

We are always looking for new and original content for our publications! Get in touch if you would like to contribute a technical article, some hi-resolution photography, or relevant company news about plastic recycling efforts.

All the best,

Did you know the SPE Foundation offers numerous scholarships to students who have demonstrated or expressed an interest in the plastics industry? Visit www.4spe.org/foundation for more information.

# 2020 Progress Report

**Editor's Note**: The following excerpts are taken from the Alliance to End Plastic Waste (AEPW) 2020 Progress Report. The full report can be downloaded via https://endplasticwaste.org/progress-report/

### It all starts with Collaboration

# RADICAL CHANGES FROM HOUSEHOLD TO COMMUNITY

Jembrana is a regency in Northwest Bali with an area of approximately 800 km2 and a population of over150,000 people. It has no formal waste management system. The waste is dumped and burnt each night and informal waste collectors pick through it to find plastic waste to sell.

Jembrana is Project STOP's (Stop Ocean Plastics) third programme in Indonesia. It is a milestone for the people of Jembrana: it is the regency's first-ever solid waste management service with a full recycling system that will create new and permanent jobs, and ensure profits stay within the community.

By 2022, the Alliance and Project STOP seek to clean up the waste that already exists and create a sustainablewaste management system that will aim to collect 20,000 tons of waste per year. 14% of this collection is expected to be plastic waste, and ultimately, 50% of this plastic waste or annual 1,500 tons per year are projected to be recycled. Project Stop Jembrana is implementing radical changes by encouraging waste segregation at the household level so that formal waste collectors can easily identify recyclable waste that can be processed at a specially-designed facility. The whole process is designed to be economically selfsufficient and fully operated by Jembrana's government and community.

#### Local needs - local responsibility

Support from the local municipal government is critical to the project's success. Foremost, the local government has provided a building for the waste collection facility, which the Alliance is supporting in terms of the design and suitability of equipment, installation and operations.

As with all projects the Alliance invests in, we do not own the resulting waste or collection business. All revenues belong to the local community and cover salaries for collectors and sorters as well as the system's operating costs. Project Stop Jembrana marks a major milestone for Jembrana – building much-needed local infrastructure by working closely with a community to make sure it is relevant to their needs. In turn, the project is formalising employment for waste collectors and providing economically suitable collection, sorting and processing systems for the people of Jembrana.

### It all starts with Stop

The project's success is possible because of the engagement and participation of communities – from operating the system to recycling at home, making sure that the right waste, in the right condition, goes into the right bin. From homes to the programme's work in schools, success also means helping to change a generation's approach to waste and recycling.

#### **Cleaning up coastlines**

The Jembrana programme was set for a test with 100 households in June 2020 before full deployment in 2021. Although COVID-19 has posed an unexpected challenge, this programme continues: the path already paved will provide effective waste management services to thousands and will keep plastic waste from leaking into Jembrana's coastlines. The aim is zero leakage of waste into the environment and growing local employment.

This is a good example of a publicprivate partnership that will deliver a replicable solution across Indonesia and beyond.

#### THE POWER OFCOLLABORATION

"Jembrana offers us the perfect entry point for ending environmental leakage of plastic in Northwest Bali. Local, regional and national government agencies are now committed partners, the community is ready for change,

and there is notable need – the majority of people have never had waste services and it will prevent thousands of tons of plastic waste from entering the environment each year. Over the next three years, together with the Alliance and.05, healthier and even more beautiful."



I Made Yudi Project STOP Programme Manager, Jembrana

### Let's All Innovate

#### CHANGING THE FUTURE OF PLASTIC WASTE MANGEMENT – AND THE PLANET

Big challenges call for new and innovative ideas. Plug and Play, as a leading innovation platform, is bringing together the best startups and the world's largest corporations to work on tackling plastic waste.

In 2019, the End Plastic Waste Innovation Platform was developed with Plug and Play to foster start-ups that can impact the plastic value chain. This accelerator programme is running across three hubs – Silicon Valley (California), Paris and Singapore – and supports ideas that transform plastic waste management.

The programme has seen more than 1,000 start-ups from Silicon Valley and Paris apply to take part, bringing with them a vast array of ideas ranging from new approaches to collecting and sorting plastic waste using optical recognition and robotics, to new chemical recycling technologies, to fresh ideas on how to use post-recycled plastics.

#### A residence-based programme

All three location-based programmes follow a similar model: each runs for 12 weeks for 10 selected start-ups and each start-up works with its own panel of seven experts drawn from Alliance member companies who offer coaching and mentorship. The end goal is for these start-ups to secure their funding through company investments and venture capital. The first to launch was the Silicon Valley programme where 20 start-ups were initially drawn from a list of 50, with a final list of 10 chosen after an in-person pitch. What followed was a 12-week residence-based programme with peer-to-peer networking opportunities and a graduation 'expo' day in May 2020. With the disruption of COVID-19, the Silicon Valley programme was extended by 90 days.

#### Looking ahead

In January 2020, we also opened the application window for the European hub of this accelerator programme, based in Paris. Due to COVID-19, all pitches have been conducted virtually. It will be extended to accommodate a residence-based programme if that becomes possible later in 2020. The Singapore hub launched in August 2020 and for now will also be a virtual-only programme. The Alliance hopes that these projects will all successfully develop over the next five years, potentially supported by investments from our member companies. In 2021, new selection rounds will start in all three hubs and we are also planning for new hubs in Asia, Africa and Latin America.

#### INNOVATIVE IDEAS THROUGH PARTNERSHIP

"The energy and excitement I have for our partnership with the Alliance to End Plastic Waste is second to none. We have had a strong start here in Silicon Valley and Paris and soon to be in



Singapore with the Alliance Members. This initiative will unite us so we can work together with more urgency. We want to accelerate these innovative ideas from great entrepreneurs and implement and invest in them."

#### **Saeed Amidi** Founder and CEO of Plug and Play

We are now studying the feasibility of two projects in Indonesia to assess if the market conditions support these large-scale infrastructure investments that convert plastic waste into a petrochemical feedstock, which is ultimately processed into fuel or high quality plastic materials.

#### Demonstrating value and economics

The first of these projects is located in Bandung, the capital of West Java, and the second is in Mojokerto, East Java.

If proven viable, these studies will result in the implementation of integrated municipal waste recovery for all waste. Engagement from local municipalities and governments for local ownership is crucial right from the beginning to ensure the success and long-term benefits of these largescale infrastructure projects.

#### Learning is key

In the absence of effective waste management infrastructure, plastic waste will end up in the environment, creating unacceptable levels of pollution. In the case of the Bandung study, which was already 60% complete before the outbreak of COVID-19, what we learn will give us guidance on the fit of advanced recycling solutions for different city archetypes.

# Advanced Recycling technologies

#### FOR THE PEOPLE, THE OCEAN AND THE LAND

Zero Plastic Waste Cities is the combined vision of the Alliance and the Grameen Creative Lab, the creative laboratory for social business solutions co-founded by Nobel Peace Laureate Professor Muhammed Yunus.

As defined by Professor Yunus, who pioneered the concepts of microcredit and microfinance, a social business model is designed to address a social problem through a financially self-sustaining business concept that is driven by its impact on society.

Zero Plastic Waste Cities aims to recycle 28,000 tons of plastic during the first five years of operations in two locations by improving and supplementing municipal waste management, repurposing collected waste and preventing it from flowing into the ocean. It will develop sustainable social businesses that improve the livelihoods of many while preventing plastic waste from escaping into the environment. The two cities initially involved in this project are Puducherry on the southeast coast of India and Tan An in the Mekong Delta region of Vietnam.

The project supports the formal engagement of waste pickers, who collect and sort recyclable waste. Once collected, the plastic can be recycled and converted into a reusable form in the material value chain. Two options are being developed for the local markets: pelletising2 plastic waste, which in turn can be used in finished goods like garden pots or building products; and using compression moulding to convert the waste into plastic boards that will eventually be used for furniture material, such as shelves, countertops or panels.

#### How it started

As an initial step, the Grameen Creative Lab conducted feasibility studies in these two cities, analysing the current value chain and waste management system to identify strengths and weaknesses. This includes determining the different stakeholders involved, such as waste pickers, and their needs.

Having the support of the local community is pivotal to the projects. This meant working closely with the government and other non-governmental organisations (NGOs) in the area and showing the tangible benefits that projects such as Zero Plastic Waste Cities can provide. It is important to engage the most impacted communities, as these efforts depend on their input.

"The strength of these projects shows the power of entrepreneurship," says Christina Jäger, a board director of the Grameen Creative Lab. Coming from an extensive background in social business and entrepreneurship, Christina is the Director of the Puducherry and Tan An projects.

#### A five-year timeline

Having identified the main stakeholders, the Grameen Creative Lab took steps towards piloting the project: developing the social business design and respective business plan as well as hiring local project managers. They are also working with NGOs in both areas, including the Auroville community in Puducherry. There are also other organisations supporting households to segregate waste streams and to raise awareness. While COVID-19 has temporarily slowed down activities on-the-ground, the project is moving forward with the pilot implementations.

Once these social businesses are established, they aim to break-even after three years of operations. The impact of the Zero Plastic Waste Cities projects will be measured by its success in removing plastic waste and improving livelihoods. The approach of creating a sustainable business model rooted in addressing social issues resonates across many other cities in line with the expectations of both the Alliance and the Grameen Creative Lab.

#### MULTIPLYING OUR IMPACT AROUND THE WORLD

"We firmly believe that social business is part of the

solution. It provides a way to organise waste management efficiently and to recognise the hard-working people in the informal sector that often remain poor and marginalised. Thanks to the support of the Alliance, we have the opportunity to build



blueprints that can be replicated all around the world multiplying its impact. We also hope that our partnership will inspire member companies to take action in their organisations."

#### **Christina Jäger**

Director, Yunus Environment Hub & Managing Partner, The Grameen Creative Lab

## Calling All Entrepreneurs

## RECYCLING AND WOMEN EMPOWERMENT IN ACCRA

The ASASE Foundation is the brainchild of three women: Hilda Addah and Patricia Agyare from Ghana, and Dana Mosora from Romania. The name ASASE comes from Asase Yaa, which means Mother Earth in Akan mythology. It represents the resilience and nurturing nature of the earth. It is a call to revere and preserve it.

The Foundation empowers women by enabling them to start their own businesses: collecting and recycling plastic waste as a source of much-needed income. It is well-known that empowering women in developing countries benefits the wider community, both economically and socially. From the outset, the Foundation has had a positive impact.

The model is simple and effective. Plastic waste is collected on the streets of underprivileged communities in Accra and sold to CASH IT! reprocessing plants by entrepreneurs. The plastic is then regrinded and sold to other companies for reuse in other products, such as household or building materials.

#### Looking towards a brighter future

ASASE's vision is clear: by 2022, all plastic packaging in Accra will be separately collected for recycling, enabling small but sustainable businesses owned and run by women entrepreneurs. The Alliance supports ASASE to dramatically increase the quantity of waste it collects: from pounds to tons and it has already set itself an ambitious target of recycling 2,000 tons of plastic each year.

ASASE's first project is CASH IT!, which was launched in 2019 with the opening of a recycling plant in Accra's Kpone Katamanso District. Since then, the Alliance has supported the Foundation with technical expertise and advice, both in terms of educating the team and consulting on improving the efficiency of processes and systems.



## Lead Technical Article

## The influence of mechanical recycling on the properties of thermotropic liquid crystalline polymer and long glass fiber reinforced polypropylene

Tianran Chen \*, Craig D. Mansfield , Lin Ju , Donald G. Baird, Craig D. Mansfield , Lin Ju , Donald G. Baird

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#### ABSTRACT

The effect of mechanical recycling on the properties of injection-molded polypropylene (PP) reinforced with thermotropic liquid crystalline polymer (TLCP) or long glass fiber (GF) has been investigated. The 30 and 50 wt% in situ TCLP and GF reinforced composites were mechanically recycled for three processing cycles, using an injection molding machine with an end-gated plague mold. The processing temperatures used in the mechanical recycling were determined using rheological and thermogravimetric analyses to minimize the degradation of polypropylene. Recycled TLCP/PP maintained its mechanical properties, and recycling had no significant influence on its morphological, thermal, rheological, and thermomechanical properties. Morphological investigation illustrated the regeneration of TLCP fibrils during the mold filling process of each recycle. By the addition of maleic anhydride-grafted polypropylene (MAPP), significant improvements in the mechanical properties of TLCP/PP without impact on recyclability were observed. In contrast, the tensile strength of 50 wt% glass fiber reinforced composite decreased 30% while the tensile modulus decreased 5% after the third recycle. Glass fiber filled polypropylene exhibited significant fiber shortening and was not able to regenerate fibers in processing. Fiber length attrition during the recycling process led to the deterioration of the mechanical properties of the recycled glass filled composites

#### 1. Introduction

A continuous increase in the usage of fiber reinforced composites has resulted in a rapid accumulation of composite material ending up in the waste stream, leading to negative environmental impacts [1,2]. The disposal of composite waste in an environmentally friendly way has become one of the most important challenges in our society. Landfill is one of the most common disposal methods, but yet this approach has become unfavorable due to restrictive environmental legislation, loss of valuable materials, and increasing processing cost [3,4]. In order to reduce the environmental impacts and to turn composite waste into valuable resources, significant efforts have been made to recycle and reuse the composite waste in the last two decades [2,5,6].

The technologies for recycling fiber reinforced composites include thermal process, solvolysis, and mechanical recycling [7–12]. Mechanical recycling consists of grinding materials into small particulates and manufacturing new parts using the ground composites [5]. Among different recycling technologies, the mechanical recycling method is favorable due to lower energy consumption, recovery of both fibers and resin, and no use of any hazardous solvents [13]. Global warming potential (GWP) of mechanical recycling of fiber reinforced composite is on average 700% lower than the solvolysis method [14].

While mechanical recycling is a cost-effective recycling method with low carbon footprint, the wide-spread application of mechanical recycling is limited due to the deterioration of the mechanical properties of recycled composites [15–20]. Because of the lower qualities of recycled composites than virgin materials, recycled materials are usually used as fillers or reinforcements in industries where the incorporation level of the reinforcement/filler is limited to under 10 wt% [5]. Eriksson et al. [21] studied the mechanical performance of in-plant recycled glass fiber reinforced polyamide 66. The recycled samples were prepared by a processing method of repeated grinding and injection molding. Mechanical analysis of recycled samples showed more than a 30% decrease in tensile strength and a 40% reduction in impact strength of the composite after eight recycling steps. Applying the Kelly-Tyson model [22] to the fiber length distributions of recycled composites gave good agreement with experimental tensile strength, suggesting that the decrease of mechanical properties was attributed to fiber shortening.

To mitigate the reduction of mechanical properties of recycled composites, Colucci and coworkers [23] produced a partially (50%) recycled glass fiber filled polypropylene composite by blending virgin composite with 100% recycled composite. The 50% recycled glass fiber reinforced composite exhibited 13% and 12% enhancements in tensile and flexural strength, respectively, compared to 100% recycled composite. The mechanical properties of 50% recycled composite, however, were still noticeably lower than those of virgin composite. Thermo-oxidative and thermal-mechanical degradations together with fiber breakage gave rise to the reduction of mechanical performance of recycled composites. The need for developing novel recyclable composites in the application of mechanical recycling becomes more urgent.

Thermotropic liquid crystalline polymers (TLCPs) represent a class of high-performance engineering thermoplastics, exhibiting high mechanical properties, light weight, excellent dimensional stability, and outstanding processability [24,25]. The tensile modulus and strength of TLCPs (i.e., up to 100 GPa and 1.5 GPa, respectively, based on drawn filaments), are competitive with the mechanical properties of glass fibers (i.e., modulus ~72 GPa, strength ~3.5 GPa) [26,27]. The density value of TLCP (~1.4 g/cm3) is much lower compared to that of E-glass fiber (2.58 g/cm3), indicating that the specific properties (tensile properties divided by the density) of TLCP are higher than the glass fiber [26,28]. A material with high specific properties will be suitable for applications such as automotive and aerospace.

In order to enhance the mechanical properties of the pure matrix, TLCPs, acting as reinforcements, were directly blended with many commodity and engineering thermoplastics (e.g., polypropylene, poly (ethylene terephthalate), poly(ether ether ketone), etc.) [29-31]. These composites were known as in situ TLCP composites because TLCP was elongated into fibrils in processing operations such as injection molding. It was reported that TLCP fibrils were formed during the blending process of TLCPs with other thermoplastics [29,32]. The dispersed TLCP in thermoplastics was deformed into fibrils by hydrodynamic forces (elongational and shear stresses). The deformation and breakup of the dispersed droplets in a polymer blend were mainly controlled by the viscosity ratio and capillary number [33]. Elongational flow was able to effectively deform the droplets over a greater range of viscosity ratios than the shear flow field.

The generated TLCP fibrils were considered to enhance the mechanical properties of thermoplastic materials. For instance, polyether sulfone (PES) was melt blended in an extruder with TLCP, and the in situ 30 wt% TLCP/ PES extruded strand showed a five-time increase in tensile modulus and a two-time improvement in tensile strength as compared to neat PES [32]. Additionally, highly oriented fibers were observed under a scanning electron microscope (SEM). De Souza and Baird [34] reported the generation of in situ TLCP composites based on the blends of partially miscible TLCP with poly(ether imide) (PEI) using the injection molding process. At 30 wt% TLCP reinforcement, the tensile modulus of TLCP/PEI was 8.7 GPa, where the tensile modulus of neat PEI was only 3 GPa. SEM demonstrated the skin-core morphological structure of TLCP/PEI composite. The elongational flow from the advancing front led to the formation of fibrils in the skin layer.

In addition to comparing TLCP composites to pure matrices, it is important to evaluate the properties of in situ TLCP composites as compared to conventional glass fiber reinforced materials. Bafna et al. [35] compared the mechanical properties of PEI reinforced with 30 wt% TLCP to short glass fiber reinforced PEI. The composites were prepared by the injection molding process. The tensile modulus of in situ TLCP composite (9.8 GPa) was found to be competitive with glass fiber reinforced PEI (9.2 GPa) at same fiber weight fraction. The tensile strength of TLCP composite (152 MPa) was not as high as that of the glass filled composite (170 MPa). Handlos et al. [29] performed injection modeling of 20 wt% TLCP reinforced poly(ethylene terephthalate) (PET) and compared its properties to those of a 20 wt% glass fiber/PET. The tensile modulus and tensile strength of TLCP/PET were 8.87 GPa and 96.6 MPa, respectively, and 20 wt% glass fiber/PET composite exhibited the tensile modulus and strength of 7.02 GPa and 107.8 MPa, respectively. Thus, the potential of in situ TLCP composites to compete with glass fiber reinforced thermoplastics has been demonstrated.

To further enhance the mechanical performance of TLCP reinforced composites, compatibilizing agents were used to promoting interfacial adhesion of incompatible blends such as polypropylene (PP) and TLCP [36,37]. The poor adhesion between PP and TLCP was mitigated by the addition of maleic anhydride-grafted polypropylene (MAPP). MAPP enhanced the interfacial adhesion and reduced the interfacial tension, leading to a finer dispersion and uniform distribution of TLCP. O'Donnell and Baird [38] found that the addition of MAPP to TLCP/ PP blends resulted in a significant improvement in both modulus and strength as compared to those of uncompatibilized blends. At 30 wt% TLCP with addition of MAPP, the tensile modulus of the composite increased from 3.0 GPa to 4.1 GPa, and the tensile strength improved from 19.7 to 37.6 MPa. Compatibilized TLCP/PP exhibited decreased interfacial tension and higher adhesion, which resulted in a fiber dispersion of TLCP within the matrix and higher mechanical performance.

Even though extensive investigations have been carried out on the processing of TLCP reinforced composites, only a few studies involved the recycling of TLCPs or their composites [39–41]. Bastida et al. [39] reported the effect of mechanical reprocessing on the structure and mechanical properties of pure TLCPs. Despite the overall tendency of decreasing the tensile properties with increasing number of reprocessing cycles, the tensile modulus and strength of TLCPs did not change significantly within the third recycling step. The influence of mechanical recycling had a stronger effect on the mechanical properties of copolyesteramide TLCP than copolyester TLCP due to structural changes coupled with the decrease in molecular weight and degree of crystallinity.

To investigate the influence of mechanical recycling on the properties of in situ TLCP reinforced composite, two recycling routes were utilized to prepare recycled in situ TLCP/polycarbonate (PC) [40]. One was first recycling neat TLCP resin, and then recycled TLCP was blended into virgin PC. The other was the direct mechanical reprocessing of the TLCP reinforced PC composite up to four times. The recycled TLCP/virgin PC maintained the tensile modulus regardless of the number of recycling steps and only decreased in tensile strength at the first recycling step. In comparison, significant decreases in both the tensile modulus and strength of recycled TLCP/PC have been seen. The decrease in the performance of recycled TLCP/ PC composite mainly ascribed to the degradation of polycarbonate rather than the TLCP.

To recover TLCP from a TLCP filled composite, Collier and Baird [41] developed a novel reclamation process to recycle TLCP. This process consisted of reactive extrusion and selective dissolution to separate TLCP and PP with the absence of organic solvent. More than 70 wt% TLCP could be reclaimed from the PP matrix with a purity of 97%. When the neat TLCP was partially replaced with recycled TLCP, no loss in mechanical properties of injection-molded in situ TLCP/PP composites was observed.

In view of the importance of developing recyclable composite and extremely scarce research work on recycling TLCP composites, it is of great interest to study the recyclability of TLCP/PP composites using mechanical recycling. It remains unknown whether TLCP/PP has greater recyclability than glass fiber reinforced polypropylene. In order to explore the potential of TLCP composite material in reducing composite waste and replacing glass fiber composite in a variety of applications, the recyclability and performance of TLCP composite must be compared against the benchmark glass fiber filled polypropylene. In this work, we report a systematic investigation of the influence of mechanical recycling on the mechanical, thermal, morphological, rheological, and thermomechanical properties of in situ TLCP/PP and long glass fiber reinforced polypropylene. The number of recycles is selected to be three because the properties of TLCP do not change much within the third recycling step [39]. The processing temperature of recycling TLCP/PP was optimized to minimize the degradation of polypropylene using rheological and thermogravimetric analyses. It was also desired to determine whether MAPP could enhance the mechanical properties of TLCP/PP without influencing the recyclability.

#### 2. Experimental

#### 2.1. Materials

The TLCP used for this study is a copolyesteramide consisting of 6- hydroxy-2-naphthoic acid (60 mol%), terephthalic acid (20 mol%) and aminophenol (20 mol%). The TLCP is provided by Celanese and is referred to as Vectra B950. The TLCP has a density of 1.4 g/cm3 and a melting point around 280 °C [42]. Long glass fiber (GF) reinforced polypropylene pellets were supplied by SABIC with 30 and 50 wt% of reinforcement. The pellets are 8 mm long and contain glass fibers of similar length. Injection molding grade polypropylene (PP) with a commercial name Pro-fax 6523 was supplied by LyondellBasell, which has a density of 0.9 g/cm3. Maleic anhydride-grafted polypropylene (MAPP) was provided by Chemtura.

#### 2.2. Mechanical recycling of the composites

The mechanical recycling of the composites was achieved by multiple injection molding and grinding processes. The content of the reinforcement was selected to be 30 or 50 wt%. Pellets of GF/PP were dried in a vacuum oven at 80 °C for at least 24 h. End-gate plaques, approximately 80 mm long by 76 mm wide by 1.5 mm thick, were injection molded using a BOY 35E machine. GF/PP pellets were injection molded with the manufacturer's recommended processing conditions, where the barrel temperature was set at 250 °C and the mold temperature was 60 tC [43]. A granulator (Cumberland/John Brown D-99050) was used to shred the injection-molded plaques into fine particulates. The particulates of GF/PP were dried again in a vacuum oven at 80 °C for 24 h before re-injection molding and regrinding. This process was repeated three times and each reprocessing cycle was designated as re-0, re-1, re-2 and re-3, where re-0 was the first processing step involving the virgin material.

To recycle the TLCP reinforced composite, TLCP and PP (or PP/ MAPP) were first blended in a single screw extruder at 290 °C with a 1- inch diameter screw and L/D = 24. The blend material was extruded through a 3 mm capillary die (L/D = 20), and then the composite strand was guenched in a water bath and pelletized into around 6 mm long pellets. The mechanical recycling of in situ TLCP filled composites was carried out in the same recycling scheme as glass fiber reinforced composite except for the processing temperature which was optimized to be 290 °C. The blend containing MAPP was prepared by extruding PP with MAPP in the single screw extruder at 250 °C; and then PP/MAPP was pelletized and mixed with TLCP in the same extruder at 290 °C. The MAPP content in TLCP/PP/MAPP was kept as 10 wt%. Specimens were randomly selected from recycled composites for various characterization tests.

## **2.3.** Thermal stability measurements of polypropylene at processing temperature

A rheometer (TA Instruments ARES-G2) was used to analyze the thermal stability of PP at 250, 290 and 300 °C using the small amplitude oscillatory time sweep mode. PP resin was dried in a vacuum oven at 80 °C for 24 h before running the rheological measurements. Pellets were loaded between parallel plate fixtures at the designated temperature under nitrogen. The complex viscosity was monitored over time with 1% strain and 10 rad/s angular frequency. Isothermal thermogravimetric analysis (TA Instruments Q50) was performed on polypropylene resin at 290 and 300 °C under the nitrogen atmosphere. The sample was heated up from room temperature to the testing temperature at 20 °C/min. Then weight loss of the specimen was tracked as a function of time.

#### 2.4. Mechanical properties

Approximately 75 mm long by 8 mm wide strips were cut along the flow and transverse directions from the injection molded end-gate plaques. All tensile properties of the composites were measured by an Instron mechanical tester (Model 4204) with a 5 KN load cell. The cross- head speed was maintained at 1.27 mm/min, and the deformations of the specimens were measured with an extensometer (MTS 634.12). The tensile properties of each material were calculated by the average properties of at least five samples.

#### 2.5. Fiber length measurement

To characterize the fiber attrition in recycling process, composite samples were burned in a muffle furnace at 500 °C, and a mat of glass fibers remained. A small portion of fibers was extracted from the mat of fibers with a needle coated by a thin layer of epoxy. The epoxy was burned off in the furnace at 500 °C. Lengths of fiber were characterized by scanning fibers with a desktop scanner, and then were measured using image analysis software (ImageJ). More than 1000 of glass fibers were measured. Details of the fiber length measurement procedure were described in the reference [44].

2.6. Micro-mechanical modeling

To investigate the structure-property relationship of glass fiber composite after recycling, micro-mechanical modeling was utilized to predicate the tensile modulus and strength of recycled GF/PP composites. The stiffness of recycled composites was predicated using the Cox- Krenchel model [45,46]. The modified Kelly-Tyson model was used to estimate the tensile strength of recycled fiber reinforced composites [22, 47,48].

#### 2.7. Morphological properties

The morphologies of recycled GF/PP, TLCP/PP/MAPP, and TLCP/PP were examined by scanning electron microscope (SEM). Specimens were fractured along the transverse

direction in the liquid nitrogen, and then sputter coated with 10 nm thick gold. The fracture surface of each sample was characterized by a LEO (Zeiss) 1550 SEM with an accelerating voltage of 5 kV.

# 2.8. Thermal properties (differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)

A DSC (TA Instruments Discovery) was used to examine the thermal properties of recycled composites. Under nitrogen atmosphere, samples were subjected to a heat/cool/heat cycle. Samples were first equilibrated at 30 °C for 5 min and then heated up to 250 °C at 10 °C/min. The materials were cooled down to - 50 °C at- 10 °C/min, and then heated back to 250 °C at 10 °C/min. The glass transition temperature ( $T_{\rm g}$ ), melting temperature ( $T_{\rm m}$ ), and enthalpy of melting ( $\Delta H_{\rm m}$ )) were determined by a TA Instruments TROIS software.

Thermal stabilities of recycled composites were analyzed by thermogravimetric analyzer (TA Instruments Q50) under nitrogen environment with a heating rate of 10 °C/min and a temperature range of 50–700 °C. The samples were equilibrated at 50 °C for 5 min before starting the heating ramp.

#### 2.9. Rheological properties

Rheological tests were carried out at 290 °C under nitrogen using a rheometer (TA Instruments ARES-G2) to investigate the influence of mechanical recycling on the viscosity of composite blends. The small amplitude oscillatory frequency sweep mode was used with an angular frequency range from 0.5 to 500 rad/s and 1% strain.

#### 2.10. Thermo-mechanical properties

To investigate how mechanical recycling affects the thermo- mechanical properties of the injection-molded plaques, dynamic mechanical thermal analysis (DMTA) was performed using a rheometer (ARES-G2). Rectangular strips were cut from injection-molded plaques along the flow direction, and samples were clamped between rectangular torsion fixtures. DMTA was carried out in a nitrogen atmosphere with a heating rate of 2 °C/min, and a temperature range of °50 to 170 °C was selected.

#### 3. Results and discussion

## **3.1.** Optimization of the processing temperature of TLCP/PP composites

The thermal stability of polypropylene at elevated temperature is examined, because, in order to generate in situ TLCP reinforced polypropylene, PP is directly melt blended with TLCP at the processing temperature which is above the melting point of the TLCP ( $T_{\rm m}$ ~280 °C) [42,49]. A parallel plate rheometer was operated in the small amplitude oscillatory time sweep mode under nitrogen. The evaluation of complex viscosity,  $|\eta^*|$ , of polypropylene as a function of time is shown in Fig. 1.  $|\eta^*|$  of PP gradually decreases with time at 290 and 300 °C. The residence time of overall recycling processes using the injection molding machine is around 12 min  $|\eta^*|$  of PP decreases by ~52% in 12 min at 300 °C (Fig. 1). The thermal degradation of PP through chain scission leads to a decrease in viscosity. The rate of decrease of the  $|\eta^*|$  at 300 °C is much more rapid than at 290 °C (Fig. 1). Blending PP with TLCP at 290 °C can mitigate the severe thermal degradation of polypropylene. 250 °C is the manufacturer's recommended injection molding processing temperature of GF/PP pellets. At this temperature,  $|\eta^*|$  of PP stays constant, thereby suggesting that the thermal degradation of PP is negligible, as shown in Fig. 1 [43]. The processing temperature of recycling GF/ PP is selected to be 250 °C.

In addition to characterizing the thermal degradation of polypropylene by rheological analysis, isothermal TGA was used to investigate the thermal stability of PP at 290 and 300 °C, as indicated in Fig. 2. Under a nitrogen atmosphere,



Figure 1. The change of complex viscosity  $(|\eta^*|)$  of polypropylene as a function of time under a nitrogen atmosphere.



Figure 1. The change of complex viscosity ( $|\eta^*|$ ) of polypropylene as a function of time under a nitrogen atmosphere.

the weight loss of polypropylene at 300 °C is about 3%, and the weight change at 290 °C is minimal, within 12 min. At 300 °C, the weight loss of polypropylene resin indicates the formation of volatile degraded products, which may result in the poor mechanical properties of the final products. Ballice and Reimer [50] investigated the thermal degradation of polypropylene, and the organic volatile products generated from thermal degradation were identified with gas chromatography/ mass spectrometry. A noticeable degradation of PP began at 300 °C. The major volatile organic compounds evolved from the thermal decomposition of polypropylene were 2-methyl-4-octene, 2-methyl-2-octene, 2–4 dimethyl-1heptene, etc. The emission of volatile organic compounds may deteriorate the performance of materials. Based on

the results of isothermal time sweep rheological test and TGA, the injection molding processing temperature of TLCP filled composites is selected to be 290 °C.

#### 3.2. Tensile properties of recycled composites

Tensile tests of the first injection-molded (re-0) composite samples are carried out to assess the mechanical performances of these composites. The tensile properties of TLCP/PP/MAPP, TLCP/PP, GF/PP, TLCP, and PP in the flow direction are presented in Table 1. By blending TLCP or GF with polypropylene, significant enhancements in the tensile properties of the composites are observed. The stiffness of composites is improved by six-fold with the addition of 50 wt% TLCP or glass fiber. The reinforcements also effectively enhance the tensile strengths of the composites. The tensile strength of the composite is improved 114% by blending 50 wt% TLCP. The incorporation of 50 wt% long glass fiber into polypropylene enables more than three times enhancement in the tensile strength of the composite. Glass fiber improves the tensile strength of the PP composite more than that of TLCP/PP composite. GF has a higher tensile strength (~3.5 GPa) than that of TLCP (~0.5 GPa) resulting in the difference in the level of reinforcement of injection molded composites [51,52]. 30 wt% TLCP and glass fiber also enhance the mechanical properties of polypropylene as shown in Table 1.

Polymer	Modulus (GPa)	Specific Modulus (Pa/(kg/m <sup>3</sup> ))*10 <sup>°6</sup>	Strength (MPa)	Specific Strength (Pa/(kg/ m <sup>3</sup> )) *10 <sup>^4</sup>
50 wt% TLCP/ PP/MAPP	8.21 (±0.255)	7.46	53.09 (±1.91)	4.83
5350 wt% TLCP/PP	$(\pm 0.23)$ ( $\pm 0.23$ )	6.47	43.96 (±1.64)	4.00
50 wt% GF/PP	7.84 (±0.37)	5.89	83.54 (±1.98)	6.29
30 wt% TLCP/ PP	3.83 (±0.13)	3.80	30.64 (±0.66)	3.03
30 wt% GF/PP	5.21 (±0.34)	4.65	71.62 (±5.55)	6.39
TLCP	17.53 (±1.16)	12.52	130.5 (±8.05)	9.32
РР	1.23 (±0.06)	1.37	20.5 (±0.31)	2.28

Table 1. Tensile properties of the initial injection molded materials (re0) in the flow direction.

Table 1 includes the specific tensile property of each material. The specific property was calculated through dividing the tensile property of each material by its density. By adding TLCP or glass fiber, the specific tensile properties of the polypropylene composites are increased appreciably. Materials with high strength-to-weight ratios have been widely employed for automotive and aerospace applications. The density of glass fiber is almost two times higher than those of the TLCP. The specific modulus of 50 wt% TLCP/PP/MAPP composites is 27% higher than that of glass fiber reinforced polypropylene, and the specific tensile strengths of 50 wt% TLCP/PP/MAPP are 17% lower than those of 50 wt % GF/PP.

To evaluate the performance of TLCP/PP composites in flow direction, the tensile modulus is predicted by the Halpin-Tsai equation [53]. The equation is simplified by assuming an infinite fiber aspect ratio. The experimental tensile moduli of injection-molded pure TLCP and PP are used in the prediction. The tensile modulus of 30 wt% and 50 wt% TLCP/PP is lower than the predicted tensile modulus (~4.75 GPa and ~7.6 GPa, respectively). The



Figure 3. Tensile properties of recycled TLCP/PP/MAPP, TLCP/PP, and GF/PP versus number of recycle steps in flow direction (a) tensile modulus; (b) tensile strength.

Halpin-Tsai model assumes perfect adhesion between fiber and matrix, which is most likely not true for the TLCP and PP due to the incompatibility between the polymers.

To overcome the problem of incompatibility and poor adhesion and further improve the mechanical properties of TLCP reinforced polypropylene, maleic anhydride-grafted polypropylene (MAPP) is introduced. MAPP is able to enhance the interfacial adhesion of the blend and improve the compatibility between PP and TLCP [36]. From Table 1, the addition of MAPP increases the tensile modulus by 15% and the tensile strength by 21% as compared to the uncompatibilized 50 wt% TLCP/PP. In addition, the tensile modulus (~8.21 GPa) of TLCP/PP/MAPP is higher than that predicated by the Halpin-Tsai equation (~7.6 GPa). MAPP reduces the interfacial tension, leading to a finer dispersion, better adhesion and uniform distribution of TLCP, which improves the mechanical properties [38]. The comparison of morphological properties between TLCP/PP/MAPP and TLCP/PP will be discussed in the morphological subsection.

The mechanical properties of recycled TLCP/PP/MAPP, TLCP/PP, and GF/PP (re1 to re3) in the flow direction are included in Fig. 3. After three reprocessing cycles, 50 wt% TLCP/PP/MAPP and TLCP/PP retain their tensile modulus and strength. Repeated mechanical recycling has no negative impact on the tensile properties of the TLCP filled composites. 30 wt% TLCP/PP also maintains tensile properties after recycling. The in situ TLCP/PP blend is capable of generating TLCP fibrils, where dispersed TLCP droplets are elongated into fibrils during polymer processing operations especially when the process involves elongational flow [29]. During the injection molding process, TLCP fibrils are generated by elongational flow at the advancing front [54]. The generation of highly oriented TLCP fibrils at each recycling process enables the recycled composite with the level of reinforcement that is similar to virgin composite. Additionally, the regeneration of TLCP fibrils eliminates fiber breakage issue which has been commonly observed in the processing of glass or carbon fiber reinforced composites. The morphological characterization of TLCP/PP composites illustrates the regeneration of TLCP fibrils, which will be discussed in the morphological subsection.

On the other hand, mechanical recycling exerts a strong influence on the mechanical properties of glass fiber filled polypropylene, in which the tensile strength of 50 wt% GF/PP drops by 30% and the tensile modulus decreases from 7.8 to 7.4 GPa after the third reprocessing cycle. The tensile modulus and strength of 30 wt% GF/PP decrease from 5.2 to 4.5 GPa and 71.6 to 44.9 MPa, respectively. It is speculated that the decrease in the tensile properties of GF/PP with the increasing number of processing cycles is attributed to fiber attrition. In the composite theory, the aspect ratio (L/D) of fiber affects the mechanical properties of composites. Long fiber reinforced composites are able to achieve higher mechanical performance than short fiber reinforced composites [55]. Decreasing of fiber length in the final product usually decreases the stiffness, strength, and impact properties of composites [56]. Fiber length and micro-mechanical modeling are used to investigate the structure-property relationship of recycled GF/PP, which will be discussed in the next subsection.



Figure 4. Tensile properties of recycled TLCP/PP/MAPP, TLCP/PP, and GF/PP versus number of recycle steps in transverse direction (a) tensile modulus; (b) tensile strength.

Injection-molded end-gate plaques provide the information of properties of specimens in both flow and transverse directions. Fig. 4 demonstrates the mechanical performance of recycled composites in the transverse direction. TLCP filled composites show little or no change in the tensile modulus and strength during three recycling steps. TLCP/PP/ MAPP exhibits higher mechanical properties than TLCP/PP composites in the transverse direction. Thus, MAPP can enhance the mechanical properties of TLCP/PP in the flow and transverse directions. For the 50 wt% glass filled polypropylene, the tensile modulus decreases by 14% and the tensile strength drops from 41.4 to 24.5 MPa after three reprocessing cycles. Mechanical recycling also leads to a significant decrease in mechanical properties of 30 wt% GF/PP in transverse direction (Fig. 4).

Overall, the mechanical properties of TLCP reinforced composites are not influenced by recycling, but mechanical recycling has a strong negative effect on the mechanical properties of glass fiber filled polypropylene. The influence of mechanical recycling on the recycled 30 wt % GF/PP or 30 wt% TLCP/PP is very similar to those of 50 wt % GF/PP or 50 wt% TLCP/PP as shown in Figs. 3 and 4. The following discussions only compare the properties of 50 wt% TLCP/ PP to GF/PP composite.

#### 3.3. Fiber length and micro-mechanical modeling

After the injection molding process, fibers in the final part are not uniform in length. To investigate the influence of fiber attrition on the mechanical properties of glass filled composite, fiber length distributions of recycled GF/PP were quantified. The procedure of fiber length measurement is described in the experimental section. Fig. 5 shows the cumulative frequency of fibers as a function of fiber length of recycled 50 wt% glass fiber composites. The initial fiber length of glass fiber is around 8.0 mm. Severe fiber breakage occurs during the first injection molding cycle (re0), in which more than 90% of the fibers are shorter than 2 mm. Repeated injection molding and grinding lead to a much narrower distribution as compared to re0-GF/ PP. For instance, 90% of the fibers of re3-GF/PP are shorter than 0.55 mm. Table 2 lists the number average (Ln) and weight average (Lw) of the fiber lengths of recycled GF/ PP. Just one injection molding process results in the Lw of 1.83 mm. Ln and Lw keep decreasing with the increase of recycling number. In the 3rd reprocessing cycle, the reduction in the Lw is 74% as compared to re0-GF/PP. There is considerable fiber attrition during melt processing due to fiber-polymer interaction, fiber-fiber interaction, and fiber-processing equipment interaction. Researchers have observed the fiber shortening when recycling glass fiber/ thermoplastic composites using mechanical reprocessing [18,19,21,57].

Table 2
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Number and weight average fiber leng	gths (mm) of recycled 50 wt% GF/PP.
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No. Recycle	0	1	2	3
$L_n$ (mm)	0.792	0.487	0.412	0.346
$L_w$ (mm)	1.829	0.644	0.548	0.465

Table 2. Number and weight average fiber lengths (mm) of recycled 50 wt% GF/PP. h.



Figure 5. Fiber length distributions of recycled 50 wt% glass fiber reinforced polypropylene.

By utilizing fiber length information of recycled GF/ PP, tensile modulus and strength in flow direction are predicted by the Cox- Krenchel and the modified Kelly-Tyson model, respectively [22,45, 55]. In Fig. 6, experimental tensile data are compared with computational modeling. Micro-mechanical models show good agreement with experimental results. Predicted tensile modulus and strength follow a similar trend that the properties increase with increasing fiber length. Mechanical properties increase slowly at the short fiber length region, followed by exponential growth at intermediate fiber length and eventually a plateau at the long fiber length region. The experimental value of both tensile modulus and strength decrease with the increasing number of reprocessing cycles. However, mechanical recycling has more impact on the tensile strength than the tensile modulus of recycled composites. 50 wt% GF/PP decreases 35% in tensile strength and 5% in tensile modulus after three recycling steps. This phenomenon may be explained by the different fiber lengths at rapid change on the Cox-Krenchel and Kelly-Tyson modeling curves. For tensile modulus, the maximum rate of decrease is around 0.2 mm. The weight average fiber lengths of recycled glass fiber composite are much higher than this value. Experimental tensile data fall into the upper region of the theoretical curve of tensile modulus, where tensile modulus is less sensitive to the change of fiber length. On the other hand, the steepest decrease of tensile strength on the modeling curve is about 1.4 mm. The weight average fiber lengths of recycled GF/PP are around this fiber length



Figure 6. Micro-mechanical predictions versus experimental mechanical properties of recycled 50 wt% GF/PP.

region. Therefore, tensile strength will be significantly impacted by decreasing fiber length. The good agreement between experimental data and micro-mechanical models suggests that fiber breakage is the major factor leading to deterioration in mechanical properties of glass fiber composite.

Deviations between predictions and experimental results of re0-GF/ PP have been observed. Fiber bundles and fiber curvatures, which are not considered by the models, could reduce the effective fiber length, resulting in overpredication of mechanical properties for the first injectionmolded composite [58,59]. With the increasing number of recycling steps, the effect of fiber bundle and fiber curvature on the mechanical performance is mitigated through better dispersion of fibers and fiber shortening. Thus, theoretical predictions become more accurate for the properties of re1 to re3-GF/PP.

In conclusion, theoretical predictions are in agreement with the tensile properties of recycled glass fiber composites. Tensile modulus and strength decrease with the decreasing fiber length. Because the specific fiber length at the maximum rate of change of properties depends on the specific property, different extents of impact by recycling on the modulus and strength are observed in the experiment. The structure-property relationship of recycled GF/PP composites is successfully analyzed by micromechanical modeling.



Figure 7. SEM images of 50 wt% GF/PP and TLCP/PP. Samples were prepared by injection molding into air: (a) GF/PP (b) TLCP/PP.

#### 3.4. Morphological properties

To reveal the major factor which leads to the difference in mechanical performance of recycled composites, morphological properties of recycled TLCP and glass fiber reinforced polypropylene were analyzed by scanning electron microscopy (SEM). Fig. 7 shows the fracture surfaces of 50 wt% TLCP/PP and GF/PP. Samples were prepared by directly injecting the composite materials into the atmosphere. A large number of glass fibers are present in the glass fiber reinforced composite sample, as shown in Fig. 7(a). Conversely, TLCP forms a spherical shape in the blend, as presented in Fig. 7(b). TLCP droplets cannot effectively reinforce the matrix compared to fibrils. The formation of TLCP fibrils usually occurs under strong elongational flow. Consequently, before the TLCP blend was injected under high pressure into the mold cavity, TLCP appears to be large and undeformed droplets in the matrix, while glass fiber composite exists as a fiber-matrix structure.

In the molding filling process, complex flow fields result in a skin- core morphology of injection molded parts [38,54,60]. For fiber filled systems, the skin layer contains highly oriented fibers, aligned parallel to the flow direction due to strong elongational flow developed at the melt front. The elongational flow moves fibers from the core region to walls where fibers quickly freeze along the cold mold wall and lock down their orientations. The core region consists of transversely oriented fibers. Because as fiber/polymer blend spreads to fill the mold cavity at the initial filling process, a strong expanding flow tends to orientate fibers in the transverse direction. The SEM images of re0 and re3 of each composite are shown in Fig. 8. All the samples were fractured along the transverse direction. The skin-core morphologies have been seen in all specimens. The skin-core structures of re0-GF/PP, re0TLCP/PP/MAPP, and re0-TLCP/PP are shown in Fig. 8(a)-(b), (e)– (f), and (i)-(j), respectively. In the skin region, glass fibers align along the main direction of flow (Fig. 8(a)). The core region of re0-GF/PP with fibers oriented transversely to the flow direction is exhibited in Fig. 8(b). In Fig. 8(c) and (d), the re3-GF/PP composite shows a similar skin-core morphology.

TLCP filled composites also exhibit skin-core structures with or without MAPP. In the uncompatibilized TLCP/ PP blend, the TLCP phase forms ribbons and fibers in the skin, as indicated in Fig. 8(i). In contrast, TLCP/PP/ MAPP exhibits finer and uniform dispersion of TLCP, and individual fibrils can be identified, as shown in Fig. 8(e). The finer TLCP fibrils with more even distribution lead to higher level of reinforcement, thereby confirming that TLCP/PP/ MAPP has better mechanical properties than the TLCP/ PP composite. In Fig. 8(f) and (j), the core regions of TLCP filled composites exhibit large droplets and transversely oriented fibers.

To investigate how TLCP reinforced composites are able to maintain their tensile properties at each recycling step, morphologies of TLCP/PP before injection molding, re0-TLCP/PP, and re3-TLCP/PP are compared. As observed in Fig. 7, TLCP forms undeformed droplets before being injected into the mold. All the fibrils which appear in the injection- molded samples (Fig. 8(e) and (i)) are generated during the mold filling process. When TLCP is remelted and reprocessed using injection molding, it is able to regenerate highly oriented fibril structures, resulting in high stiffness and strength composite. Fig. 8(g) and (k) exhibit the appearance of fibrils in the skin regions of re3-TLCP/ PP/ MAPP and TLCP/PP. The appearance of TLCP fibrils in the SEM images confirms the regeneration of fibrils after recycling. In conclusion, the regeneration of fibrils enables the high level of reinforcement, and thereby recycled TLCP reinforced composites are as strong as virgin material. However, recycled glass fiber filled composite suffers fiber breakage and is not able to regenerate fibers during the injection molding process, leading to the loss of mechanical performance.

#### 3.5. Thermal properties

DSC and TGA were used to examine the thermal properties of recycled materials. Because the thermal behavior of TLCP/PP/MAPP and TLCP/PP is very similar, the following discussion will focus on comparing the thermal properties of TLCP/PP/MAPP and GF/PP. Figs. 9 and 10 show the second heating and first cooling curves of recycled 50 wt% GF/PP and TLCP/PP/MAPP. Samples were first heated to 250 °C at 10 °C/min, cooled down to -50 °C at -10 °C/min, and reheated from 0 to 250 °C at 10 C/min. The upper temperature limit of DSC was selected to be 250 °C to avoid any thermal degradation of polypropylene during experiments and damaging the machine. In Fig. 9, the second heating curves of re0 and re3 GF/PP display similar glass transition temperatures ( $T_{g}$ ) of PP at -12 °C. The glass transition of polypropylene, in recycled TLCP filled composites, is also around -12 °C shown in Fig. 9. The glass transition temperature of PP in both composites is unaffected by the recycling numbers. The endothermic peak, at 160 °C, is corresponding to the melting point  $(T_m)$ of polypropylene (Fig. 9). There is no significant change of  $T_{\rm m}$  as a function of the increasing number of reprocessing cycles for both composites. From the first cooling scans of recycled composites, the exothermic peaks of GF/PP and TLCP/PP/MAPP are 120 °C and 117 °C, respectively as shown in Fig. 10. No change in Tc was observed after three reprocessing cycles. The degrees of crystallinity (Xc) of GF/ PP and TLCP/PP/MAP were calculated from the area under the melting endotherm with respect to the enthalpy of 100% crystalline polypropylene ( $\Delta$ H0m =207 J/g) [61]. The Xc values of re-0 GF/PP and re-3 GF/PP are 44% and 42%, respectively. The Xc of re-0 TLCP/PP/MAPP is 47% and re-3 TLCP/PP/MAPP is 46%. There is only a slight fluctuation in Xc with the increase of recycling numbers. The influence of the degree of crystallinity on the mechanical properties of recycled composites is negligible.



Figure 8. SEM micrographs of skin and core of recycled 50 wt% GF/PP, TLCP/PP/MAPP and TLCP/PP. Samples were injected into mold (a) skin re0-GF/PP; (b) core re0-GF/PP; (c) skin re3-GF/PP; (d) core re3-GF/PP; (e) skin re0-TLCP/PP/MAPP; (f) core re0-TLCP/P/MAPP; (g) skin re3-TLCP/PP/MAPP; (h) core re3-TLCP/PP/ MAPP; (i) skin re0-TLCP/PP; (j) core re0-TLCP/PP; (k) skin re3-TLCP/PP; (l) core re3-TLCP/PP.



Figure 9. DSC second heating curves of recycled 50 wt% GF/PP and TLCP/PP/MAPP

Overall, the DSC results illustrate that mechanical recycling has no significant effect on the thermal properties of recycled glass fiber or TLCP reinforced composites.

TGA is utilized to characterize the thermal decompositions of recycled GF/PP and TLCP/PP. The weight loss (%) of each sample as a function of temperature is plotted in Fig. 11. Under a nitrogen environment, the single-step degradation is observed for re-0 GF/PP, where the 5% weight loss occurs at 438 °C. This weight loss is due to the thermal degradation of polypropylene. TLCP reinforced composite decomposes in two steps. The first weight loss occurs around 459 °C, attributed to the degradation of polypropylene. The decomposition of TLCP gives rise to the second weight loss. First maximum decomposition rates of GF/PP and TLCP/PP/MAPP occur at 489 °C and 496 °C, respectively. The thermal stability of TLCP filled composites is slightly higher than glass fiber reinforced polypropylene. The TGA curves of re3- TLCP or GF composites almost overlap with re0 composites, suggesting that recycling has no particular effect on the thermal degradation behaviors of TLCP or glass fiber reinforced composites. From DSC and TGA results, multiple mechanical recycling does not significantly change the thermal behavior of glass fiber and TLCP reinforced polypropylene.

#### 3.6. Rheological properties

To characterize the rheological behavior of recycled glass fiber and TLCP reinforced composites, small amplitude



Figure 10. DSC cooling scans of recycled 50 wt% GF/PP and TLCP/PP/MAPP.

oscillatory frequency sweep tests were carried out. The complex viscosity ( $|\eta^*|$ ) is plotted as a function of angular frequency ( $\omega$ ) for 50 wt% GF and TLCP filled composites with different recycling numbers, as shown in Fig. 12. Because TLCP/PP/MAPP and TLCP/PP show similar rheological behavior, the figures include the comparison between TLCP/PP/MAPP and GF/PP. In Fig. 12(b), TLCP/ PP/MAPP exhibited weak dependence of  $|\eta^*|$  on  $\omega$  at low frequency, and the correlation between complex viscosity and angular frequency becomes stronger with increasing frequency. For GF/ PP, the complex viscosity plateau at low frequency is not observed, but rather the  $|\eta^*|$  increases significantly with decreasing  $\omega$  as indicated in Fig. 12(a).



Fig. 10. DSC cooling scans of recycled 50 wt% GF/PP and TLCP/ PP/MAPP.

This suggests the interactions of fiber-fiber and fiber-matrix increase the resistance to flow at low frequency. At high  $\omega_{i}$ these interactions are disrupted, and, thus,  $|\eta^*|$  of GF/PP melt decreases rapidly with increasing  $\omega$  [62]. By comparing the  $|\eta^*|$  of re0-GF/PP and re0-TLCP/PP/MAPP, the  $|\eta^*|$ of glass fiber reinforced composite is found to be more than an order of magnitude higher than that of the TLCP composite in the low frequency region. It is well known that TLCPs exhibit low melt viscosity even comparing to many thermoplastics [63]. Glass fibers increase the viscosity of polymer blends, reducing the processability [64]. With the increase in the number of reprocessing cycles,  $|\eta^*|$  of TLCP and glass fiber reinforced composites keep decreasing. Mechanical recycling has a stronger impact on the reduction of the complex viscosity of glass filled composite than that of the TLCP composite, especially in the low frequency region, because of the joint influence of fiber attrition and degradation of the matrix of the glass fiber composite. The decrease of  $|\eta^*|$  of the recycled TLCP composite is due to the thermal and mechanical degradation of polypropylene and TLCP during the recycling process [39,65].

#### 3.7. Thermo-mechanical properties

Dynamic mechanical thermal analysis (DMTA) was performed on injection-molded TLCP and glass fiber reinforced composites to determine the influence of recycling on the thermo-mechanical properties. There is no significant difference between the DMTA data of 50 wt% TLCP/PP/MAPP and TLCP/PP. DMTA curves of recycled TLCP/PP/ MAPP are compared against recycled GF/PP composites. Fig. 13 illustrates the storage modulus spectra of re-0 to re-3 TLCP/PP/MAPP and GF/PP composites. The storage modulus indicates the rigidity of viscoelastic materials. The storage moduli of recycled TLCP/PP/MAPP composites decrease with increasing temperature, and several rapid declines in storage modulus are associated with thermal transitions of polypropylene and TLCP (Fig. 13(b)). The storage moduli of re-0 to re-3 TLCP/PP/MAPP overlap with each other, suggesting that TLCP reinforced composite retains its stiffness at various temperatures after several reprocessing cycles. Conversely, the storage moduli of recycled GF/PP only overlap when the ambient temperature is below the  $T_{g}$  of polypropylene, and a noticeable difference is observed between the storage moduli of recycled glass fiber composites at elevated temperature as shown in Fig. 13(a). In the glassy

state, polymer chains of polypropylene are frozen, and the fiber length will not affect the stiffness of GF/PP. At elevated temperature, recycled GF/PP composites exhibit considerable reductions in stiffness. With the onset of segmental motion of the matrix polymer, the effect of fiber length on the stiffness of the composites is magnified. After three recycling steps, GF/PP decreases more than 37% of its stiffness at 100 °C. Due to the fiber breakage during recycling, shorter fibers may partially lose the ability to restrict the movement of polypropylene and decrease the overall stiffness of the composite [66,67]. Mechanical recycling imposes a significant impact on the thermalmechanical properties of glass fiber filled composite and has no influence on the mechanical performance of TLCP reinforced composites even at elevated temperatures.

#### 4. Conclusion

In situ TLCP reinforced polypropylene composites maintain high performance after mechanical recycling. The incorporation of maleic anhydride-grafted polypropylene into TLCP/PP composites has been found to significantly enhance the mechanical properties without influencing the performance after reprocessing, and the mechanical properties of TLCP/PP/MAPP are competitive with the mechanical properties of long glass fiber reinforced composite. Generation of TLCP fibrils during the mold filling process at every recycling step allows the same level of reinforcement, leading to no loss of mechanical performance. In contrast, recycled glass fiber reinforced composites show lower tensile properties, especially tensile strength (-30%), as compared to virgin glass fiber composite. Fiber breakage during recycling results in the decrease in mechanical properties of GF/PP. Therefore, TLCP reinforced composites have greater recyclability than glass fiber filled composites.

To generate in situ TLCP composite, the processing temperature of injection molding TLCP/PP is selected to be higher than the melting point of TLCP. The processing temperature of blending them can be optimized through rheological analysis and TGA. The processing condition enables the generation of recyclable TLCP reinforced composites, which have comparable mechanical performance to long glass fiber filled propylene with the same fiber weight fraction. Mechanical recycling has no significant influence on the thermal, rheological, and thermo-mechanical properties of TLCP/PP composites.



Figure 12. Frequency sweep of recycled 50 wt% GF/PP and TLCP/PP/MAPP at 290 °C.



Figure 13. Storage modulus versus temperature of recycled 50 wt% GF/PP and TLCP/PP/MAPP

On the other hand, for glass fiber filled systems, repetitive recycling has made large impacts on all the properties, except for thermal properties. The significant difference in the performance between recycled GF/PP modeling illustrates the influence of fiber breakage on the mechanical properties of recycled GF/PP. The impact of recycling to different extents on modulus and strength is because the fiber length, in which the property has the maximum rate of change, is different for different properties. Given the greater recyclability relative to GF composite, light weight, and overall excellent performance, TLCP/PP is a promising candidate to replace glass fiber reinforced polypropylene in various applications. Being able to reuse TLCPs is not only an important cost factor but an environmental factor.

#### **CRediT** authorship contribution statement

**Tianran Chen:** Conceptualization, Methodology, Software, Investigation, Writing - original draft, Supervision, Project administration.

**Craig D. Mansfield:** Conceptualization, Visualization, Writing - review & editing. Lin Ju: Investigation, Resources, Writing - review & editing. Donald G. Baird: Supervision, Writing - review & editing.

#### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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# Plastics and the Circular Economy The Virtual Edition

The Annual Plastics and Waste Conference organised by the Australia-New Zealand Section of the Society of Plastics Engineers will be run as a series of webinars under the title of "Plastics and the Circular Economy – The Virtual Edition" and will feature 20 presentations.

The Conference will have 4 separate sessions during the week from Monday 16<sup>th</sup> to Friday 20th November 2020.

The **first Session** will be on Monday evening 16th November and will feature 6 speakers mainly from Europe including an update by Juliet Lennon of the Ellen MacArthur Foundation and the latest news on plastics recycling technology by Professor Edward Kosior from Nextek.

The **second Session** will be on Tuesday morning 17th November with 4 speakers. This morning Session will commence with a Keynote address by the CEO of APCO, Brooke Donnelly, and will include a presentation by The Alliance to End Plastic Waste represented by its CEO, Jacob Duer.

The **third Session** will be on Thursday morning 19th November with 6 speakers and will focus on advances by the recycling industry in Australia. As for last year we have invited the Assistant Minister for Waste Reduction and Environmental Management, The Hon. Trevor Evans MP, to update the audience on the plans of the Federal Government. This Session will also include Frank Lintvelt from Cleanaway and Andrew Smith from the PACT Group as well as representatives from Qenos and LyondellBasell.

The **fourth Session** will be on Friday morning 20th November and will feature at least 5 speakers from the USA including Steve Alexander, the CEO of the Association of Plastics Recyclers, and the global President of the Society of Plastics Engineers, Jaime Gomez.

In this COVID-19 year we have all had to change the way we work and work together. With supply chains being reviewed, we have an excellent opportunity to re-shape the industry in Australia and speed up the transformation to the Circular Economy, so we encourage you to make your contribution and be a part of this Conference.

For the complete program see below or our local website: www.plastics.org.au

#### Monday evening 16th November from 7 pm to 10 pm

Technology and equipment – Moderator – SPEANZ - Han Michel – President

- o Erema, Austria Fritz Wimmer Sales APAC
- Nextek, UK and Australia Professor Edward Kosior CEO
- Seabin, Australia Pete Ceglinsky CEO invited
- PRE, Belgium, "LDPE films recycling" Ton Emans President, Plastics Recyclers Europe
- Ellen MacArthur Foundation, UK Juliet Lennon Project Manager, Plastics Pact, The New Plastics Economy
- LyondellBasell, The Netherlands, for JV LYB/Suez on Quality Circular Polymers - Paula Sanabria – Project Manager Circular Economy, Global Sustainability

#### Tuesday morning 17th November from 10 am to 12 pm

Australian impact – Moderator - Kevin Thomson – SPEANZ – Chair Corporate Affairs

- o Australian Packaging Covenant Organisation Brooke Donnelly CEO
- Australian Council of Recyclers Pete Shmigel CEO
- $\circ~$  Alliance to End Plastics Waste, Singapore Jacob Duer  $\mbox{CEO}$
- $\circ~$  Waste Management and Resource Recovery Association Gayle Sloan CEO

Thursday morning 19th November from 9.30 am to 12.30 pm Local supply chain – Moderator – Steve Petinakis – SPEANZ – Member Committee of Management

- Federal Government Assistant Minister The Hon. Trevor Evans MP invited
- o Cleanaway Frank Lintvelt Strategy Manager
- o LyondellBasell Australia Mitchell Killeen Managing Director
- o **Qenos David Francis** Marketing and Technology Manager
- o PACT Group Andrew Smith, General Manager Sustainability Services
- o Visy rPlastics Australia Rob Dvorak Manager Recycling Plant

Friday morning 20th November from 8.30 am to 11.00 am Americas – Moderator – Craig Benson – SPEANZ – Councillor

- Gneuss, USA and Germany Daniel Gneuss CEO Americas
- o Society of Plastics Engineers, USA Dr Jaime Gomez President
- Association Plastic Recyclers, USA Steve Alexander CEO
- o Extended Producer Responsibility, California, USA TBN
- o Sustainability and SPE, USA Conor Carlin, VP Sustainability

For more information on the program, please contact: Han Michel, mobile 0416 168 255, email: <u>hanmichel@bigpond.com</u>

The SPE: A-NZ Section retains the right to alter and/or substitute components to the event or program as deemed appropriate. Information which we supply in relation to the event is accurate to the best of our knowledge at the time of providing this information.

## Register Now!

## **Innovation Briefs**

Trinseo collaborates with COEXPAN to validate polystyrene recycling technologies for use in the dairy industry for Form Fill Seal (FFS) market at INNOTECH

September 1, 2020 – Trinseo (NYSE: TSE), the global materials company and manufacturer of plastics, latex binders and synthetic rubber, announced today that Trinseo and COEXPAN, a global leader in sheet extrusion for FFS dairy markets in Europe, CIS and America, will collaborate to validate various polystyrene recycling technologies to develop polystyrene containing recycled content that is fit for use in the global dairy industry's form fill seal ("FFS") market. With materials and input supplied by Trinseo, Coexpan will be testing recycled polystyrene across various technologies at its INNOTECH (COEXPAN&EMSUR Innovation Technology Center for Rigid and Flexible Packaging).

The dairy FFS packaging market, which has historically preferred the use of polystyrene due to its technical superiority over alternative polymers, has recently seen greater interest in alternative materials owing to misperceptions regarding the circularity and recyclability of polystyrene.

Trinseo and Coexpan, jointly state: "r-PS is a game changer for the FFS market. Polystyrene offers a unique potential for not only closed loop recycling, but also a lower carbon footprint and extended lifecycle due to its simplicity and ease to fully recycle giving polystyrene a major competitive advantage vs all alternatives. Industrial volumes will come online by the third quarter 2022".

Dinis Mota, CEO of COEXPAN, commented: "We are very pleased to have established this collaboration with Trinseo, which will allow us to demonstrate the viability of mechanical and chemical recycling processes for Polystyrene, a polymer with excellent properties in terms of efficiency and circularity in the food packaging market category. As a global leader in FFS and thermoforming packaging solutions, working with Trinseo in this regard reinforces our joint sustainability actions." "Coexpan is very excited about this collaboration. Having seen the crucial role plastic packaging has played this year in terms of food safety and hygiene, we view recycling and circularity as even more crucial. Our research center, INNOTECH, is constantly innovating with recycling and circularity in mind. We do not launch a product without first addressing circularity. This partnership provides a unique platform that reaffirms our goal of promoting and achieving a circular economy model".

Nicolas Joly, Vice President Plastics and Feedstocks at Trinseo, added: "We are very excited about the collaboration of Coexpan and Trinseo. This marks another major milestone on the way to seeing recycled Polystyrene products on shelves. The unique recycling opportunity that Polystyrene offers is now much more broadly recognized, and it is great to be able to collaborate with leaders along the value chain to further expand this recognition. Full circularity for Polystyrene is imminent, and Trinseo is passionately committed to this, but such partnerships are key to accelerating the transition to circular economy and contributing to the European Commission's recycling goal."

Trinseo's full sustainability pledge in PS Recycling covers mechanical, dissolution and chemical technologies with a clear commitment to close the loop on PS recycling, namely:

- Announcing a first-of-its-kind polystyrene chemical recycling facility to be built in Europe;
- Planning to offer an average of 30% recycled content to customers for polystyrene packaging in Europe by 2025;
- Serving as a founding member of Styrenics Circular Solutions (SCS), a consortium that explores new methods for polystyrene recycling; and
- Partnering with the waste supply chain to help identify new ways to increase the volume and quantity of plastics disposed and recovered through waste separation.

# Why Join?

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