LATEST BREAKTHROUGHS WITH HYBRID REINFORCED COMPOSITES IN LIGHTWEIGHT APPLICATIONS

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Abstract

The shortage of landfill space, ocean pollution of plastics and the depletion of petroleum resources invigorated engineers and scientists to shift their focus on to the development of bio-based plastics. Natural fibers are abundant and provide many advantages such as: weight reduction, process-friendliness (no wear of tools & skin irritation), biodegradability and good acoustic insulation properties. The limitations of natural fibers as reinforcement for thermoplastics are their low mechanical properties, limited thermal stability and shrinkage in comparison to traditional inorganic reinforced thermoplastics. In this study, the objective was to develop hybrid composites combining poly α-1, 3-glucan (DuPont's Nuvolve™) along with long glass fiber in a polypropylene matrix to optimize the overall composite properties. The polysaccharide and glass fiber loadings (wt. %) were varied and the samples were prepared via twin-screw extrusion followed by injection molding. The results showed mechanical properties decreased with increasing polysaccharide content, however there is an optimum loading of the polysaccharide that is a promising alternative to reduce the utilization of inorganic fillers (e.g. glass fiber). Thermal & morphological analyses were also conducted to understand dispersive nature of the fillers, reinforcing capability and thermal behaviour of the overall composite. In general, the composites showed enhanced thermal stability with increasing glass fiber content with good to moderate filler distribution and dispersion within the PP matrix. The hybrid reinforced thermoplastic composites can lead to vast weight and potential cost savings with the promise to advance environmental stewardship within the automotive realm.

Introduction

Petroleum-based plastics have vastly contributed to quality of life in modern society such as the advent of Polyethylene (PE) which is widely used in consumer goods, wire & cable insulation, industrial piping, linings, coatings and was historically deployed in World War II for electrical insulation of submarines and radar shields [1]. In particular, the usage of plastics by mass in the automotive industry increased from 6% in 1970 to 16% in 2010 and it is due to reach 18% by 2020 [2]. The increased usage of plastics contributes to post-consumer waste where shortage of landfill space, ocean pollution and the depletion of petroleum resources inspired engineers and scientists to develop biodegradable & renewable plastics. Next generation materials should exemplify principles of sustainability, industrial ecology and green chemistry.

Moreover, in the United States of America the corporate average fuel economy (CAFE) regulated by the national highway traffic safety administration (NHTSA) set the fuel economy standard for passenger vehicles and light-duty trucks to be 54.5 MPG by 2025 [2]. The projected added cost to a 2025 model vehicle is 1800 USD however, the improved fuel economy for those consumers who drive their vehicle for its entire lifetime would save on average 3400-5000 USD

[2]. This standard further amplified the necessity for automakers to find innovative ways to improve fuel efficiency, where the concept of lightweighting sparked great interest. Newton's second law states that acceleration of an object is dependent on the forces acting upon the object and the object's mass, therefore it takes less energy to accelerate a lighter object. Application of this fundamental law has shown that 10% reduction in vehicle weight can improve fuel economy by 6-8% [3].

Lightweighting primarily focuses on substituting heavier objects like metal with lighter materials such as carbon fiber in high performance vehicles. This is extremely hard to achieve for large volume vehicles due to the cost and metal's structural importance in the construction of engine, chassis and body exterior parts. Metals used in non-structural parts of the vehicle can be replaced with reinforced thermoplastics without compromising performance while delivering reduction in weight. Composites are attractive combining materials properties in ways not found in nature and in the automotive industry glass fibers and talc are common materials used for reinforcing plastics due to their low cost and good mechanical properties. Naturally sourced materials such as cellulose and poly α -1, 3 glucan have potential to provide good reinforcing properties at a lower density (~1.5 g/cm^3). The advantages of bio-based filler materials are that they provide good mechanical properties, biodegradability, lower wear on processing equipment and lower density. The limitations of such materials are their restricted thermal stability, shrinkage in comparison to traditional inorganic fillers, hydrophilic nature of the filler and higher than usual cost/pound of material in comparison to inorganic fillers. Therefore, completely replacing inorganic filler material with naturally sourced material is not viable in today's economic environment, however combining inorganic and bio-based fillers could provide an intermediate and necessary solution to achieving performance properties, lightweighting and biodegradability.

Improvements to fuel efficiency are initial steps to achieving a circular economy. However, there is also a need for a holistic approach to engineering design for next generation automotive parts that consider the source of materials, processing and disposal at end of usage which all contribute to the total carbon footprint associated per vehicle production.

The purpose of this research was to develop hybrid composites combining polysaccharides of unique morphologies with glass fiber in a polypropylene matrix. In particular, DuPont's poly α -1,3 glucan (derived from sucrose feedstock) to reinforce polypropylene in combination with glass fiber for body interior and underhood applications such as IP substrate, center console and battery cover in passenger vehicles & light-duty trucks. Composites were produced at various loadings of polysaccharide and glass fiber using melt blending (twin screw extruder) and injection molding. The effect of the combination of filler loading on the composite properties were investigated (mechanical and thermal) to gain insight of strategies to optimize performance properties of the composites. This research attempts to provide a comprehensive study on the development of hybrid composites containing naturally sourced materials to deliver optimum mechanical and thermal properties suited for body interior and underhood applications for passenger vehicles and light-duty trucks while achieving significant lightweighting opportunities.

The major challenge of incorporating polysaccharides in polypropylene matrix is surface compatibility due to the hydrophilic and hydrophobic nature of polysaccharides and polypropylene. Filler-matrix adhesion is critical, as the role of the matrix in a fiber-reinforced plastic is to transfer the load to the stiff fibers through shear stresses at the interface; which requires good adhesion. It is desired to have strong binding capabilities between the filler and the matrix and having weak binding sites promotes void structures and particle agglomeration. Therefore, it is essential to maximize the interfacial compatibility of the filler within the continuous polymer matrix.

Experimental

Polypropylene (PP) homopolymer pellets, long glass fiber filled PP pellets were provided by local suppliers and poly α-1,3-glucan of two particles sizes (5 & 20 microns) were supplied by DuPont Industrial BioSciences under the tradename Nuvolve™. Polypropylene grafted maleic anhydride (PP-g-MA: locally sourced) was used as a coupling agent to help with the compatibility of hydrophobic PP and hydrophilic polysaccharide.

The composites were prepared using a two-step process i) extrusion and ii) injection molding. Nuvolve™ masterbatch (30%) with PP was compounded using twin-screw extruder (ThermoHaake Rheomex Model PTW25) and prior to extrusion all materials were dried to reduce moisture content using an oven at 60°C for 12 hours. The dry PP, PP-g-MA and Nuvolve™ were separately starve-fed into a twin screw extruder via K-Tron gravimetric feeders. After extrusion, the materials were immediately quenched in a water bath and kept at room temperature. The compounded materials from the twin screw extruder were granulated using a lab scale grinder/chopper and dried at 60°C 12 hours before the injection molding step.

Nuvolve™ masterbatch (30%) with PP (now grounded pellets) was hand-mixed with long glass fiber filled PP pellets and then transferred to the injection molder (Boy Machines Model 80M) to process ASTM test specimens for tensile (ASTM-D638-10), flexural (ASTM D790-10) and impact (ASTM D256 – 10) testing, respectively.

Table 1 shows the formulations used in this study, where two particle sizes of DuPont's Nuvolve™ (Nuv A=5 microns, Nuv B=20 microns) were supplied and the control samples used were 5% & 10% glass fiber reinforced PP, virgin PP and Ford's incumbent material (35% total filler content). It is also important to emphasize the exclusion of a second extrusion step to incorporate the glass fiber in the Nuvolve™ masterbatch (30%) with PP. The elimination of a second extrusion process can speed up part production, lower manufacturing costs and reduce thermal degradation and fiber attrition.

Table 1: Sample identification codes and composition of composites

Formulation Table				
Sample Code	**Nuvolve™ A (wt. %)**	**Nuvolve™ B (wt. %)**	**Glass Fiber (wt. %)**	**Total Filler (wt. %)**
Neat PP	---	---	---	---
5%GF_PP	---	---	5	5
10%GF_PP	---	---	10	10
10/10-A	10	---	10	20
10/15-A	10	---	15	25
10/20-A	10	---	20	30
15/15-A	15	---	15	30
20/10-A	20	---	10	30

10/10-B	---	10	10	20
10/15-B	---	10	15	25
10/20-B	---	10	20	30
15/15-B	---	15	15	30
20/10-B	---	20	10	30

Mechanical Test & Density Measurement

Tensile, flexural and impact tests were conducted using a universal testing machine (Instron 3366) and a pendulum tester (Testing Machines Inc. 43-02-03 model) in compliance with ASTM D638-10, ASTM D790-10 and ASTM D256 – 10, respectively. The properties of interest were: tensile strength, strain, young's modulus, flexural modulus, maximum flexural stress and impact resistance. All mechanical tests were run in an environmentally conditioned room at 23°C± 2°C and 50± 5% relative humidity. Density was measured using an analytical balance (readability down to 0.1mg) and density kit ME-DNY-43 from Mettler Toledo.

Thermal Characterization

Thermal transitions of the composites and the neat polymer matrix were analyzed using a differential scanning calorimetry instrument (DSC: TA Instruments Q2000). At first the samples were heated from room temperature to 190°C at a rate of 50°C/min and isothermally held for five minutes. Thereafter, the samples were cooled to 70°C at 10°C/min and isothermally held for 5 minutes before reheating to 190°C at 10°C/min. The melting and crystallization transitions were collected from the heat flow versus temperature curves where, melting temperature (T_m) is an endothermic transition and the crystallization temperature (T_c) is an exothermic transition denoted by the peak minimum and maximum respectively. Heat of fusion (ΔH_m) and enthalpy of crystallization (ΔH_c) were also calculated.

Thermal gravimetric analysis (TGA: TA Instruments Q500) was used to study thermal stability of neat PP and the composites. The samples were heated at 20°C/min and subjected to nitrogen from room temperature to 800°C and then held isothermally in air for five minutes. The degradation temperature of the resin and fillers can be identified from TGA.

Morphology (SEM)

Zeiss 1550 (LEO) scanning electron microscope with accelerating voltage of 5-7.keV was used to observe the morphology of the composites and the neat PP as well as distribution of fillers within the polymer matrix. The samples were cut using a paper cutter (guillotine) exposing fracture surface of the samples Furthermore the samples were sputter-coated with gold to avoid surface charging

Results and Discussion

Mechanical Properties

Tensile Properties & Density Measurement

The effects of different fiber combinations on the tensile properties of the composites and control samples which include neat PP, PP filled with glass fiber (5 &10%) and Ford Motor Company's incumbent material are showcased in Figure 2, 3 and 4. The composites are labelled as 10/10, 10/15 and etc. where, the first number represents Nuvolve™ content and the latter represents glass fiber content. Overall, the addition of inorganic and natural fillers led to a considerable increase in the tensile strength at maximum load and young's modulus. The inorganic filler (glass fiber) had a greater improvement in strength and modulus values compared to poly α-1,3-glucan (DuPont's Nuvolve™); an increase of 122% and 190% for tensile strength at maximum load and modulus respectively was observed for Nuvolve™/GF (10/20) composites compared to neat PP. Note that 30/0 formulation that only contained Nuvolve™ as a filler performed poorly in tensile strength (16% decrease), however improved modulus (29% increase) when compared to neat PP. It is expected that glass fiber improves tensile strength and modulus at a higher factor than naturally sourced materials due their strong and stiff mechanical properties. The strongest performer of the hybrid composites was 10/20 formulation followed by 10/15 and 10/10 formulations which utilizes 20-25% of total filler content in comparison to Ford's incumbent material (35% total filler content). Figure 1, shows the density reduction (%) compared to Ford's incumbent material that is being used for body interior and underhood applications; where formulations 10/15 and 10/20 show a density reduction of 13 and 10% respectively. Therefore, the combination of inorganic and naturally sourced filler materials can offer enhanced mechanical properties with sufficient weight savings per vehicle part.

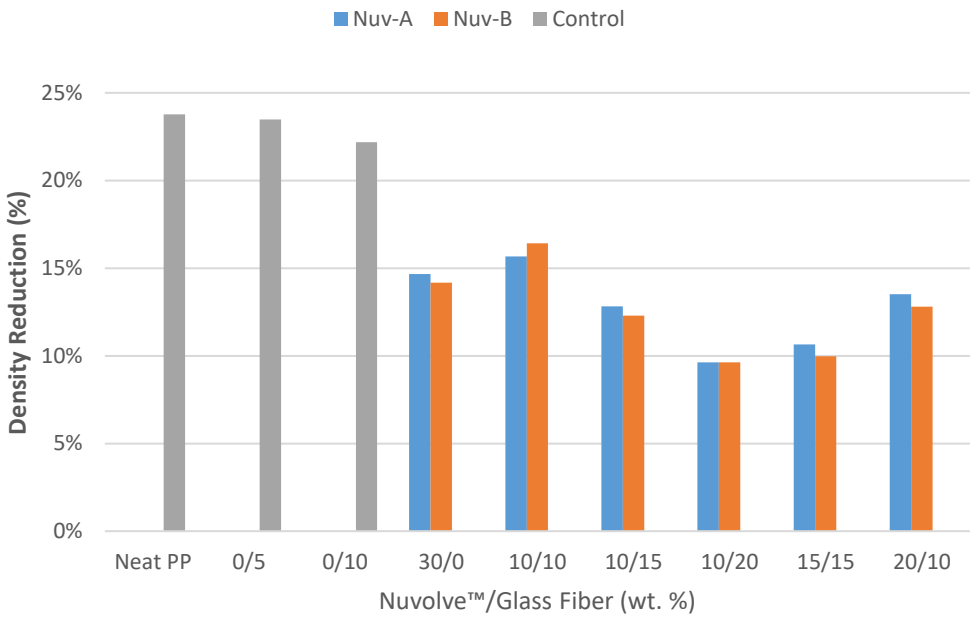

Figure 1: Density Reduction (%) of all composites and neat PP in reference to Ford Incumbent Material

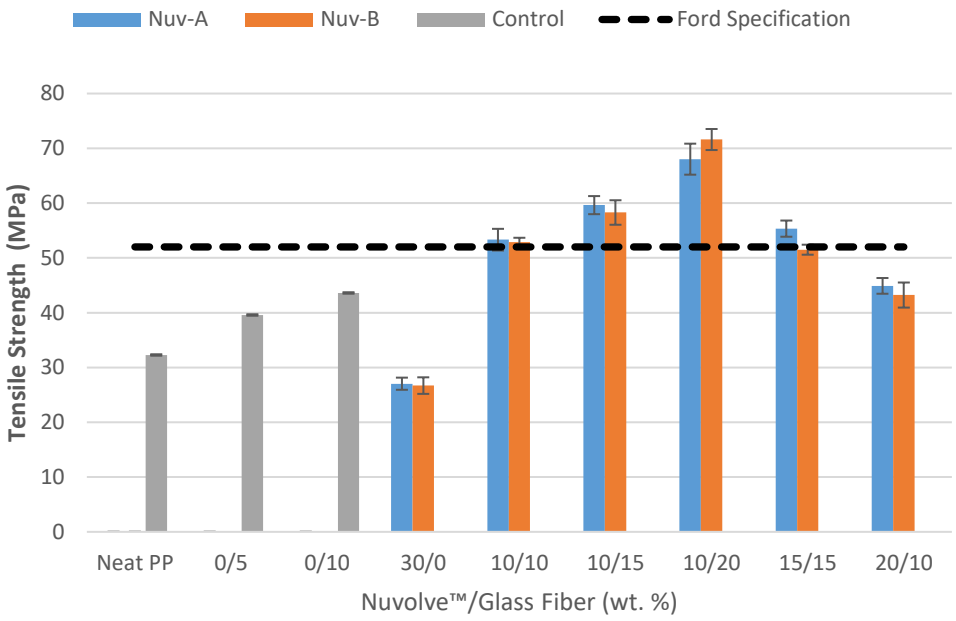

Figure 2: Tensile Strength (MPa) of composites and neat PP

The tensile strains at maximum load of the composites were lower than neat PP (Figure 4) with no considerable difference in strain as a function of total, inorganic and natural filler content.

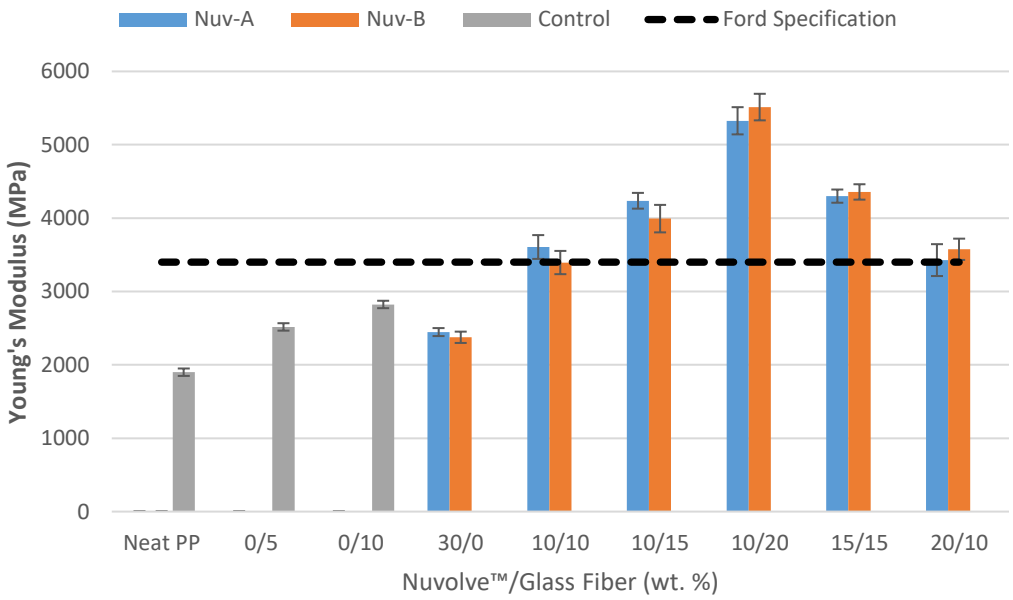

Figure 3: Young's Modulus (MPa) of composites and neat PP

Overall the tensile properties showed that there is a greater influence in performance via the addition of glass fibers compared to DuPont's Nuvolve™, however hybridization of both fillers produced beneficial enhancements in performance and lightweighting.

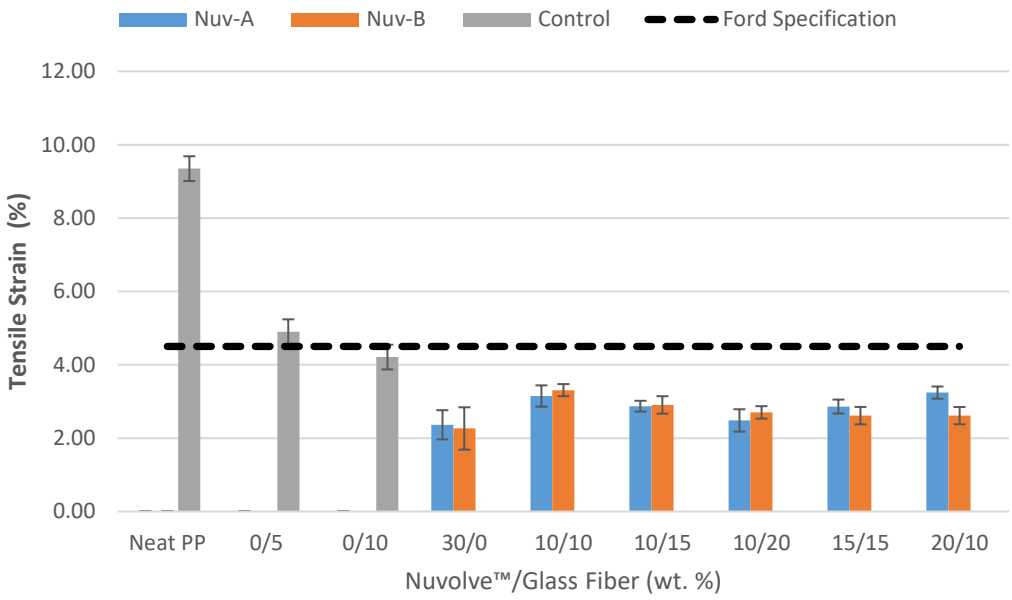

Figure 4: Tensile Strain (%) of composites and neat PP

Flexural Properties

Flexural properties exhibited a similar trend as the tensile results, showing that the addition of glass fibers had a positive impact on the modulus and maximum stress.

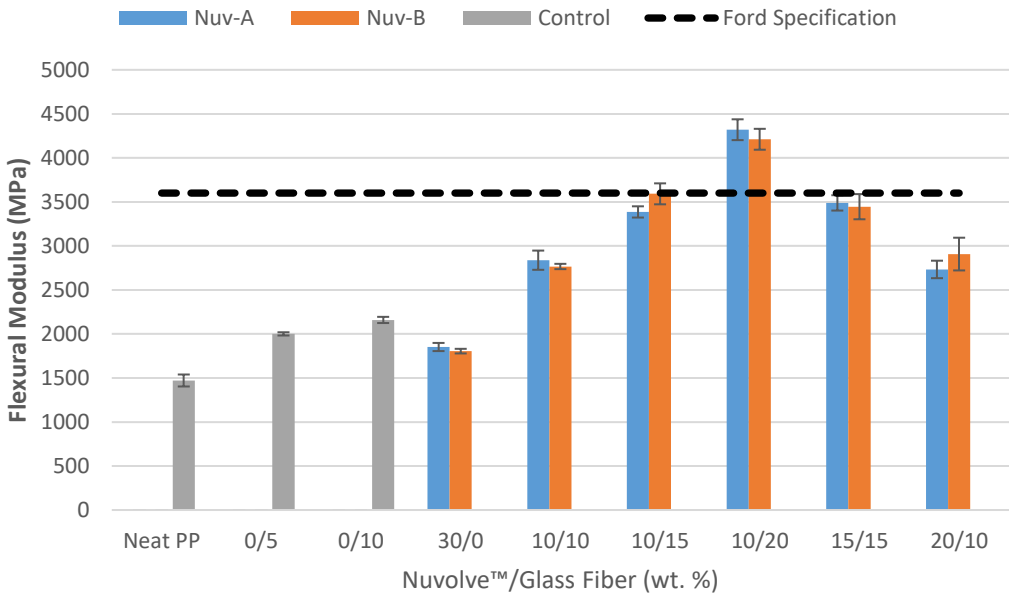

Figure 5: Flexural Modulus (MPa) of all composites and neat PP

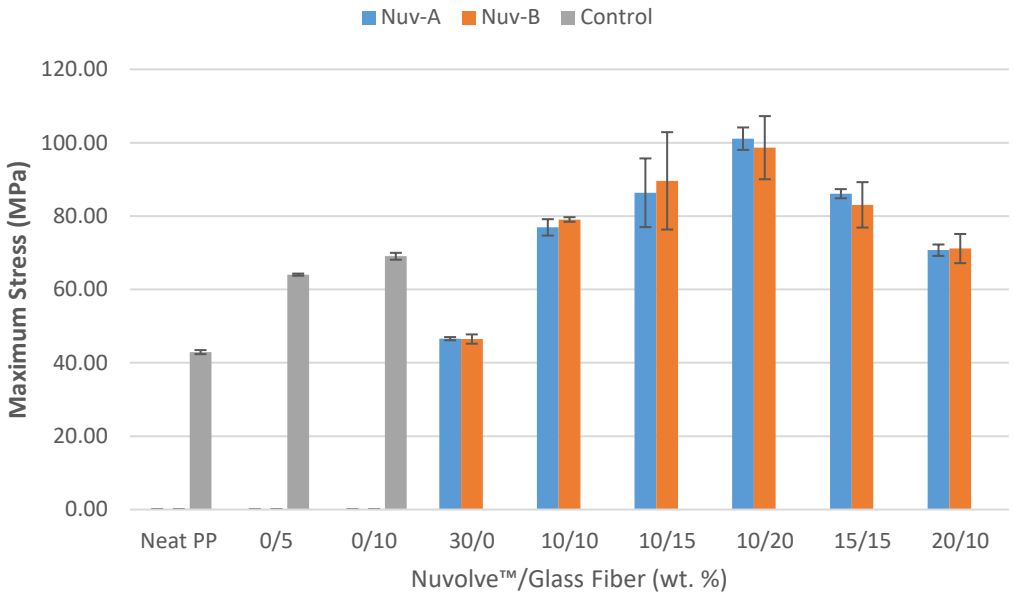

Figure 6: Maximum Stress (MPa) on all composites and neat PP

The addition of DuPont's Nuvolve™ showed a 22% increase in modulus (Figure 5) compared to neat PP and the highest performing composite 10/20 had an overall increase of 193% in modulus and 74% in maximum stress (Figure 6) compared to neat PP. Ford's incumbent material has a modulus of 3600 MPa and the 10/15 formulation was able to meet the requirement. It should be noted that there is no official requirement listed for maximum stress and hence Ford Motor Company does not specify an official value for this metric.

Impact Properties

Figure 7 shows that impact resistance decreased by 54% for formulation 30/0 and this can be attributed to the dispersive nature of the material as well as promoting stress concentrations within the polymer matrix. This is consistent with Panthapulakkal and Sain's [4] findings, where they found hemp/glass fiber PP composites had enhanced impact resistance with an increase of glass fiber content. Moreover, the hybrid composites had an increase in impact resistance up to 123% (10/20) compared to Neat PP and this is largely attributed to glass fiber content. It can also be noted that the 10/10 formulation meets and exceeds Ford's incumbent material requirement by 50% and showcases a density reduction of 17% (Figure 1). Therefore, the hybrid composites are able to provide superior impact resistance while offering lightweighting opportunities.

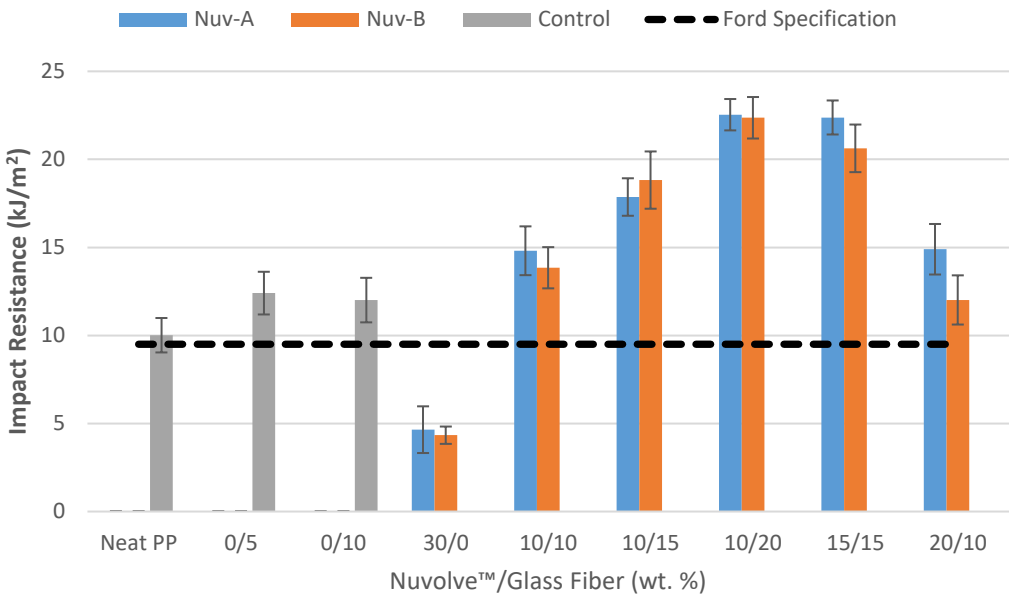

Figure 7: Impact Resistance (kJ/m²) of all composites and neat PP

Overall, the addition of DuPont's Nuvolve™ and glass fiber in a PP matrix enhanced mechanical performance. The greatest contributing factor is the concentration of glass fiber in the composite, however the addition of Nuvolve™ also provides for enhanced mechanical properties (to a lesser degree) while offering superior weight savings per material part. 10/15 formulation was the optimal solution as it was able to meet Ford's incumbent material specification with the exclusion of tensile strain. Furthermore, this formulation showed a density reduction of 13% compared to Ford's incumbent material, showcasing lightweighting opportunities in non-structural components of an automobile.

Thermal Properties

The mechanical properties showed no clear distinction in performance between Nuv-A and Nuv-B. Nuv-B has a particle size of 20 microns (4 times larger than Nuv-A) and it costs less to manufacture indicating commercial potential. Consequently thermal and morphological data are presented with respect to Nuv-B. Figures 8, 9, 10 and 11 summarizes the thermal stability of the composites and neat PP, demonstrating the onset decomposition temperature of Nuvolve™ & Polypropylene (T_{dN}, T_{dP}) and temperatures at 10% & 15% weight loss from TGA curves. The crystallization & melting temperatures are presented in Figures 12 & 13 along with values for heat of fusion and crystallization in Table 2.

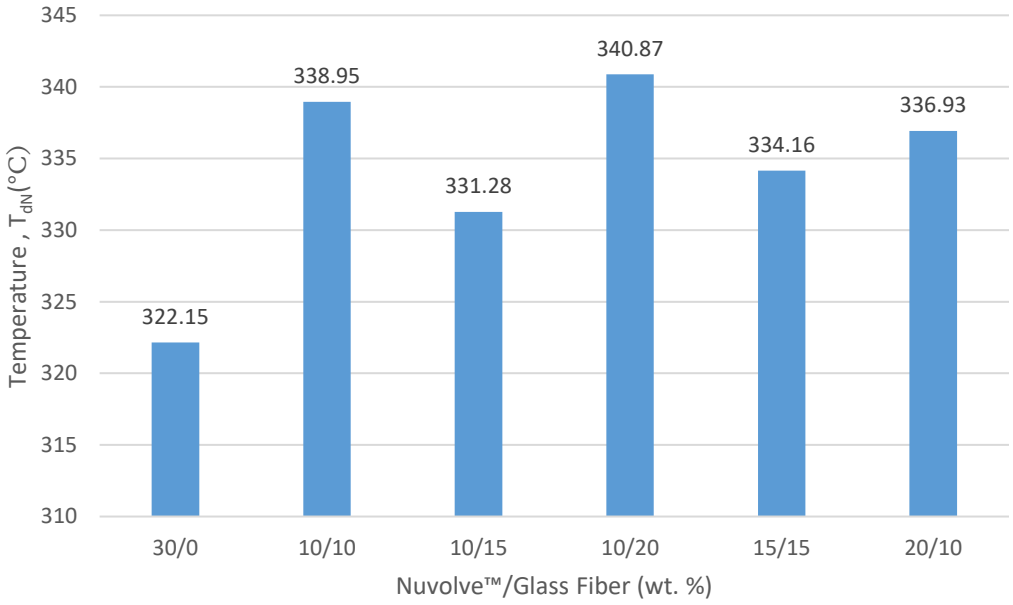

Figure 8: Onset Decomposition Temperature of Nuvolve™ A

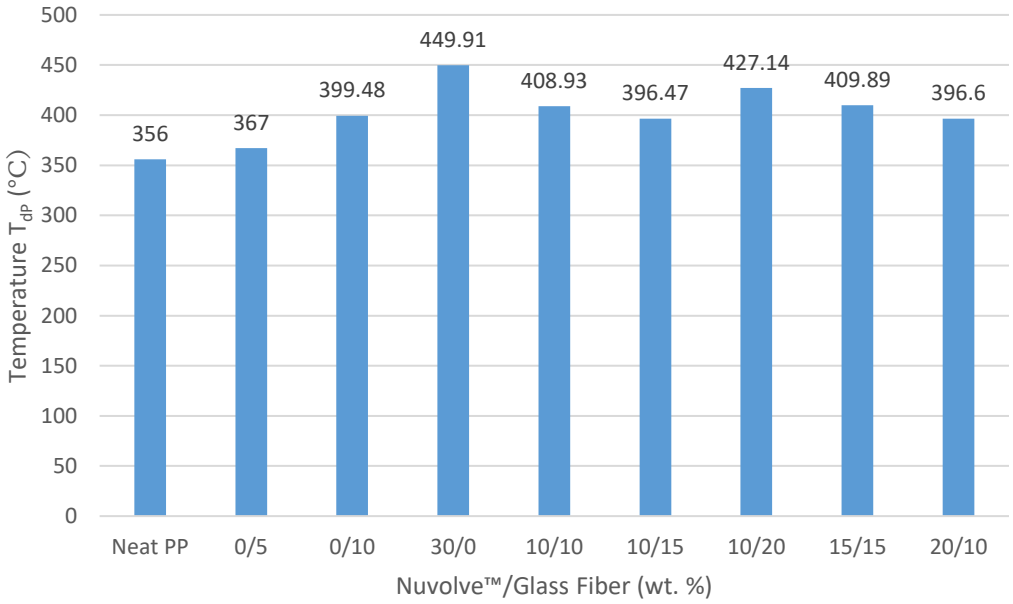

Figure 9: Onset Decomposition Temperature of PP

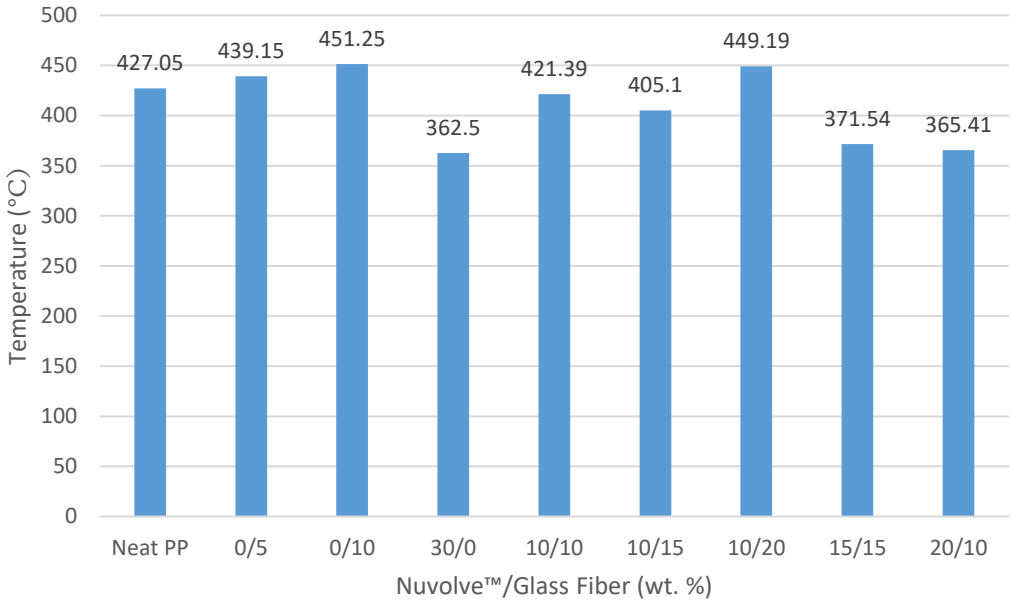

Figure 10: Temperature at 10% weight loss from TGA curves

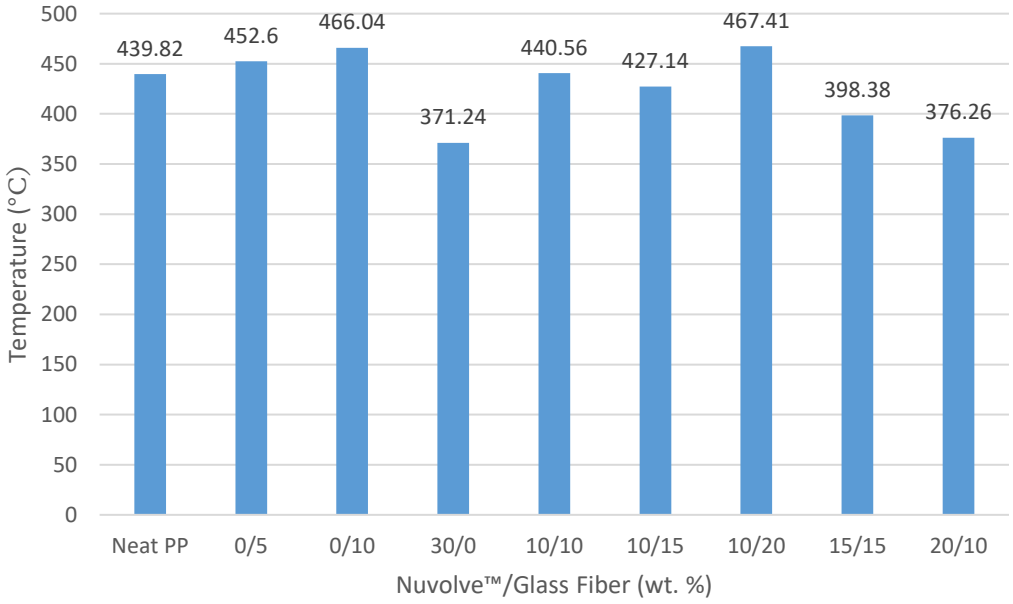

Figure 11: Temperature at 15% weight loss from TGA curves

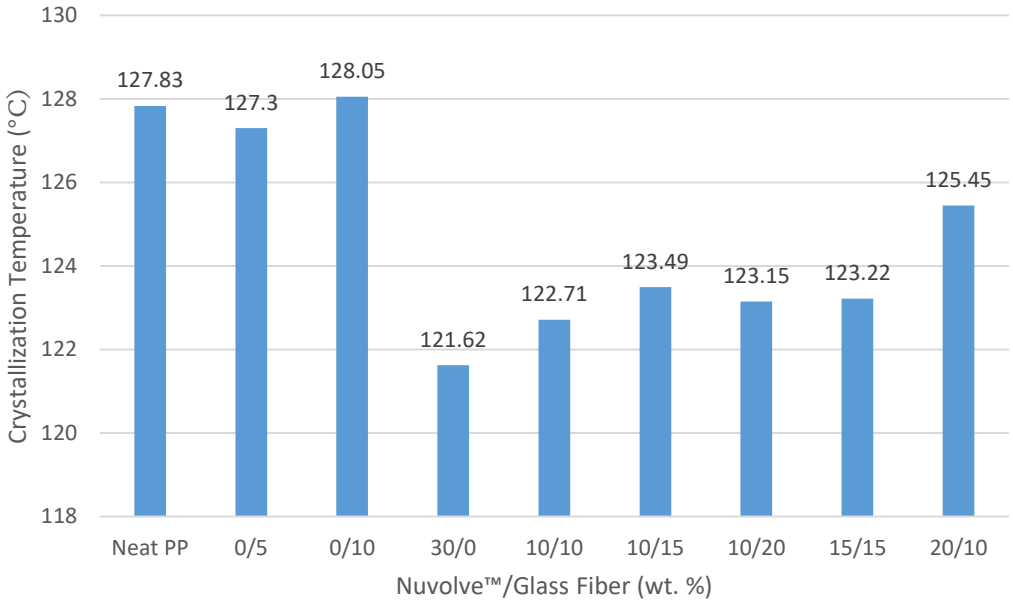

Figure 12: Crystallization Temperature (T_c)

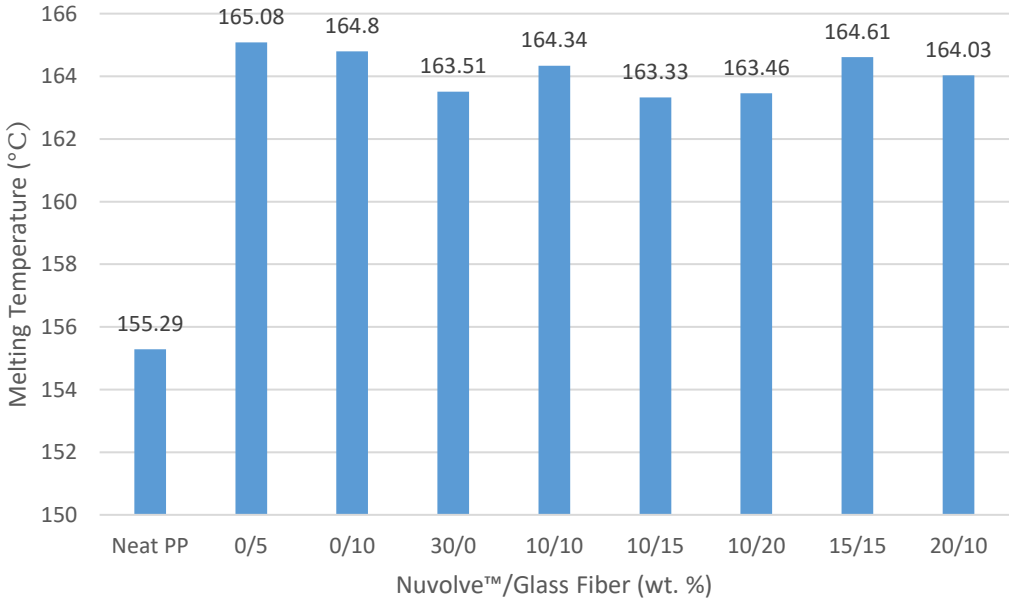

Figure 13: Melting Temperature (T_m)

Table 2: Enthalpies of crystallization and melting for neat PP and composites

Enthalpies from DSC		
Sample Code	**Heat of Fusion ΔH_f (J/g)**	**Enthalpy of Crystallization ΔH_c (J/g)**
Neat PP	139.20	129.9
0/5	100.70	98.72
0/10	143.40	129
30/10	81.11	91.99
10/10	106.90	115.2
10/15	96.86	102.1
10/20	91.11	97.31
15/15	103.50	102.71
20/10	99.83	102.9

Figure 8 shows that the decomposition of Nuvolve™ is consistent and does not fluctuate, formulation 30/0 consisting of only Nuvolve™ showed the lowest value and there is a slight positive correlation with the addition of glass fiber in the composites (5.8% increase). The onset degradation temperature of PP gradually increased, formulation 30/0 showed a 26% increase with respect to neat PP. Moreover, the temperatures at which 10% and 15% weight loss (Figures 10 & 11) occurred were also used to evaluate thermal stability. The hybrid composites were more stable than neat PP with the glass fiber having a greater influence. Formulation 30/0 showed a 15% decrease in temperature compared to neat PP.

Figures 12 and 13 summarize the crystallization (Tc) and melting temperatures (T_m) of the control samples and the composites. The crystallization temperature decreased for all composites with respect to neat PP. This could outline the possibility of the Nuvolve™ and the glass fiber not being able to act as nucleating agents to aid in faster crystallization of the PP. On average there is a 3% decrease in crystallization temperature of all composites relative to neat PP. The melting temperature increased for all composites in comparison to neat PP (5% increase), however the effect of Nuvolve™ is minimal on the melting temperature which stayed consistent for all composites.

Overall, the composites showed enhanced thermal stability in comparison to neat PP. The integration of Nuvolve™ did not profoundly increase thermal stability of the composites, glass fiber had a greater influence. However, combining both materials showed that the composites were thermally stable (compared to neat PP) with the upside of potential cost effectiveness in regards to weight savings per automotive part. The DSC results showed that the fillers do not act as nucleating agents to aid with accelerated crystallization which translates to a potential increase in part production and reduction in cycle times. The melting temperature increased for all composites compared to neat PP but, there was minimal differences across the composites alluding to the little influence of individual fillers on the melting temperature. Table 2, shows the enthalpies of crystallization and melting and the general trend observed is that the composites have decreased values compared to neat PP when incorporating Nuvolve™ as opposed to glass

fiber. Generally, the thermal properties show that the hybrid composites can be utilized in challenging conditions e.g. underhood applications offering thermal stability.

Morphological Properties

Formulations 10/10, 10/15 and 10/20 were the best performers with respect to mechanical and thermal properties and therefore are demonstrated in this section. Figures 14, 15, 16, 17 and 18 show SEM images at 100x magnification of neat PP and the hybrid composites with the following filler loading: 30/0, 10/10, 10/15 and 10/20.

Figure 15 shows that Nuvolve™ is evenly distributed and dispersed in the continuous PP matrix, where no clear distinction can be concluded to separate filler from matrix. Figures 16 and 17 show good distribution of glass fiber in regards to formulation 10/15 and 10/20, therefore the reinforcement capability is maximized as seen in their respective mechanical performance. In Figure 19, the micrograph shows Nuvolve™ without the addition of glass fiber in the PP matrix and it can be observed that the poly α-1, 3-glucan forms agglomeration of particles with an average size of 30 microns which translates to poor transfer of load between filer and matrix. Furthermore, the dark shadows around the particles indicate poor adhesion between the Nuvolve™ and the continuous polymer phase.

Figures 19, 20, 21, 22 show micrographs where glass fiber can be distinguished from Nuvolve™ by their difference in diameter and shape (Figure 20). Overall good dispersion and moderate distribution is observed for the hybrid composites. Poly α-1, 3-glucan and polypropylene are hydrophilic and hydrophobic by nature, and therefore it is difficult to attain good adhesion at the interface between the filler and matrix. Good to moderate adhesion is directly related to the composite's mechanical performance as the filler's sole duty is to effectively transfer load from the polymer matrix.

Figure 14: SEM micrograph of Neat PP at 100x magnification

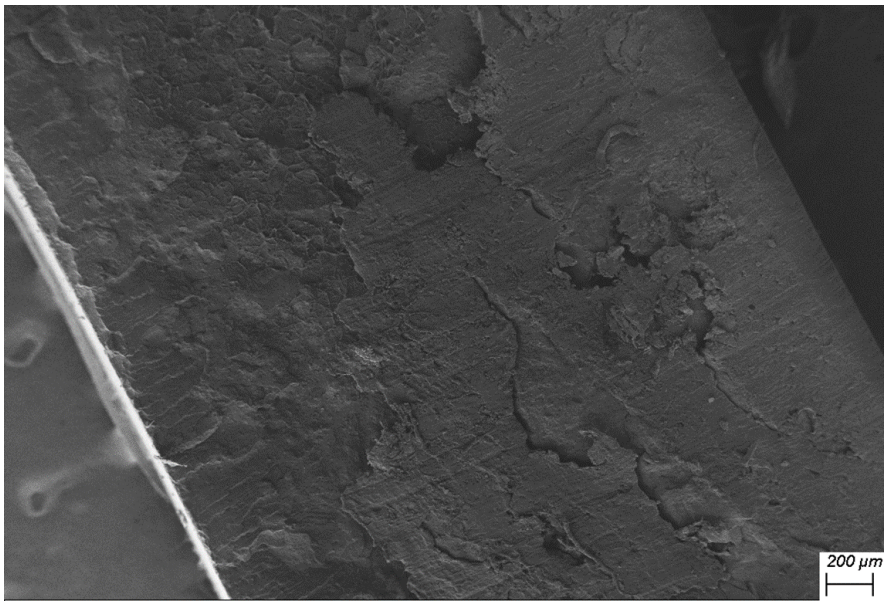

Figure 15: SEM micrograph of 30/0 formulation at 100x magnification

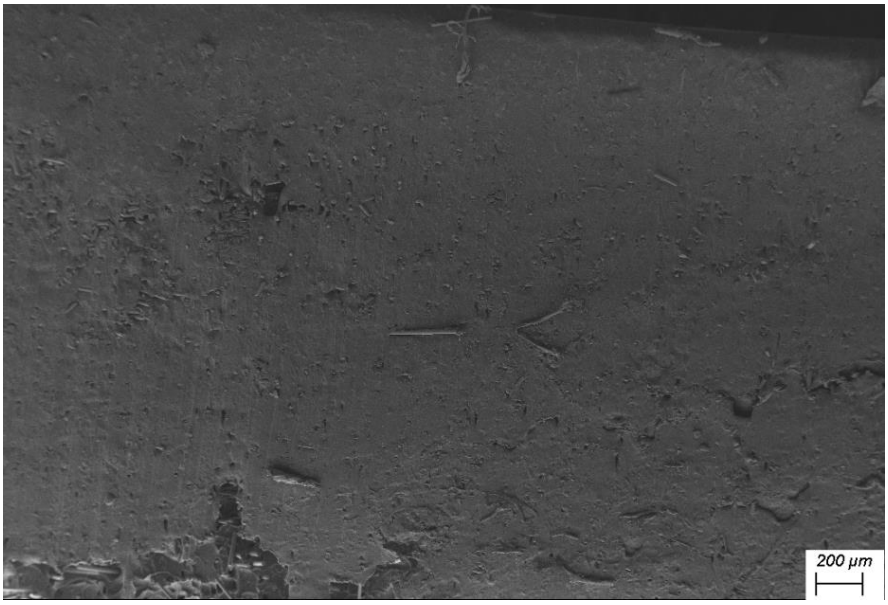

Figure 16: SEM micrograph of 10/10 formulation at 100x magnification

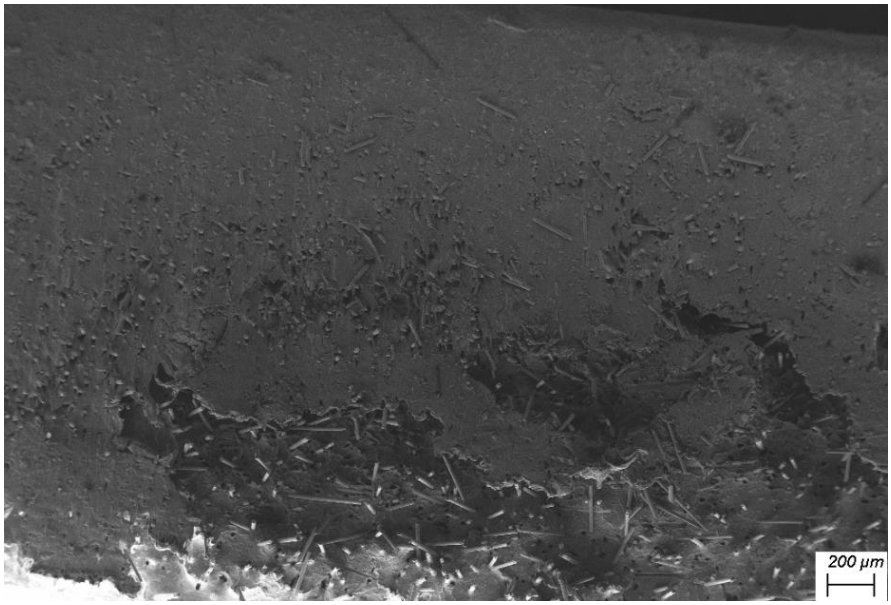

Figure 17: SEM micrograph of 10/15 formulation at 100x magnification

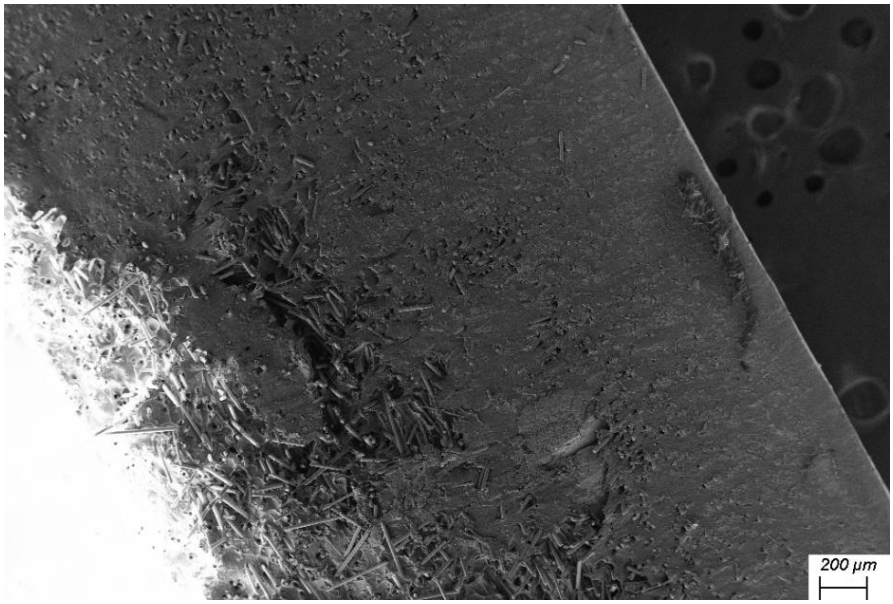

Figure 18: SEM micrograph of 10/20 formulation at 100x magnification

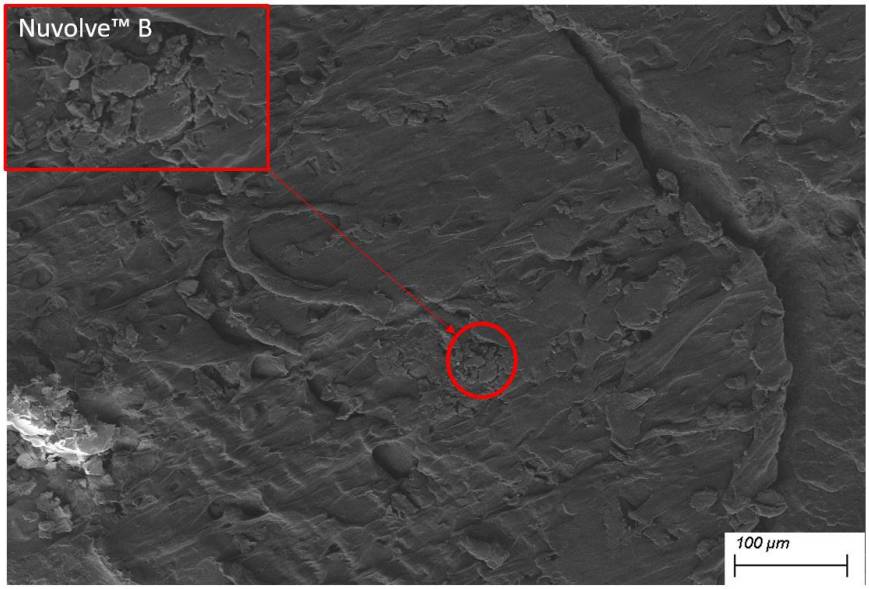

Figure 19: SEM micrograph of 30/0 formulation at 500x magnification

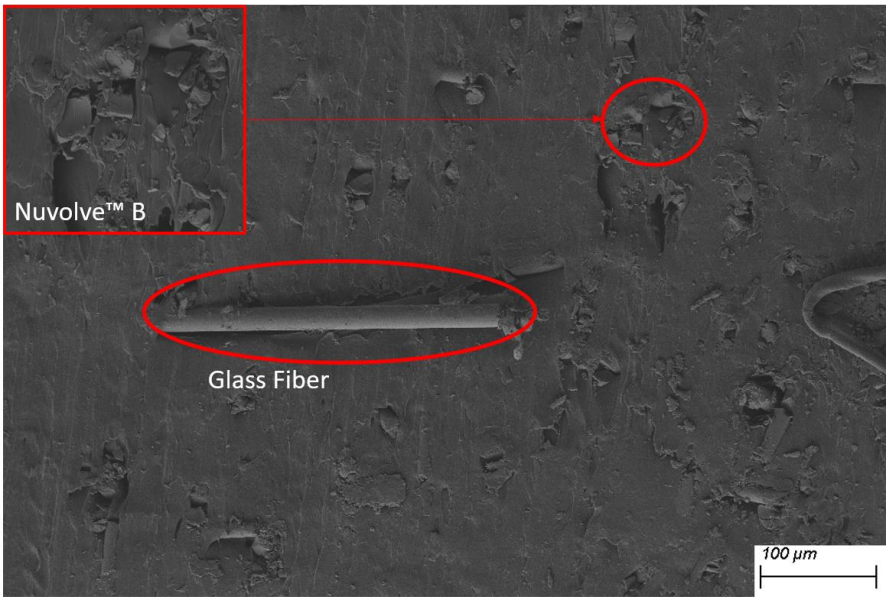

Figure 20: SEM micrograph of 10/10 formulation at 500x magnification

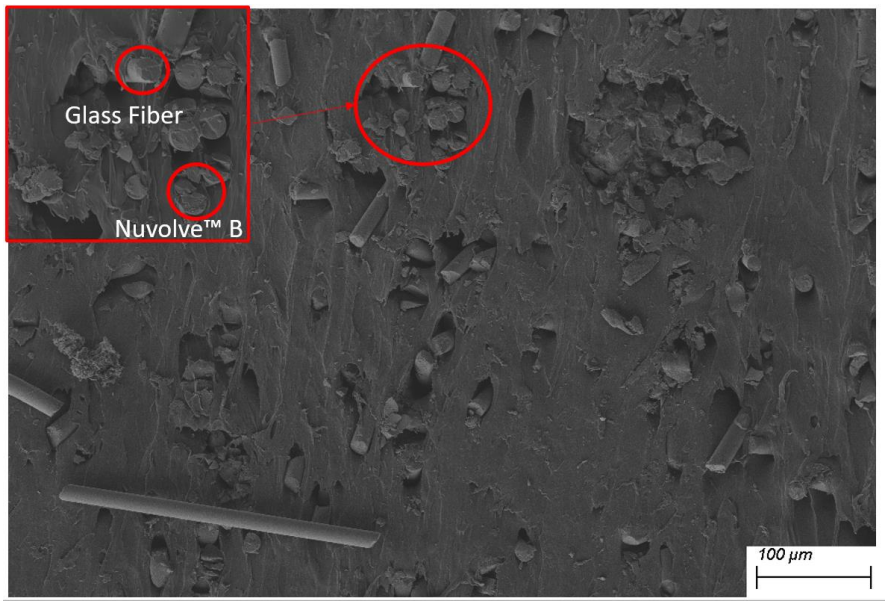

Figure 21: SEM micrograph of 10/15 formulation at 500x magnification

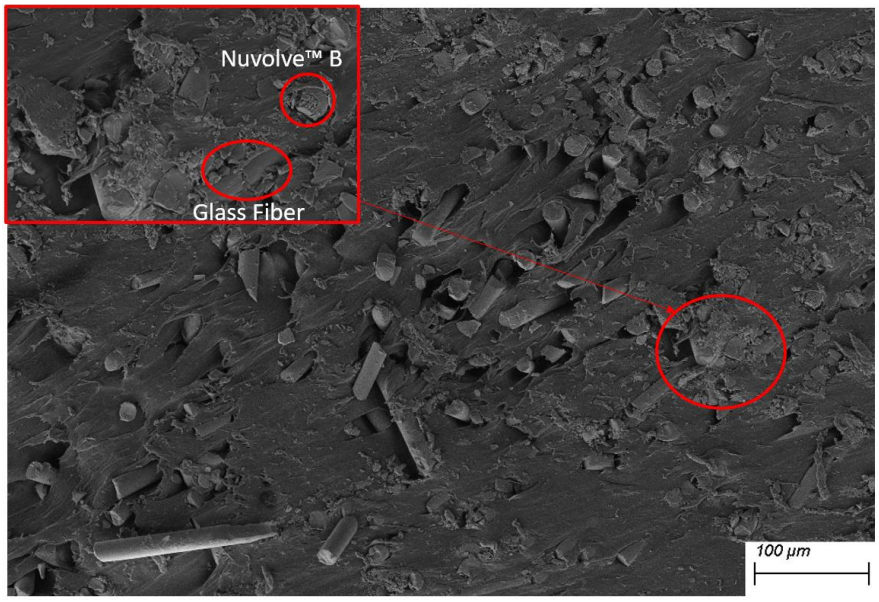

Figure 22: SEM micrograph of 10/20 formulation at 500x magnification

In Figure 21, fiber pull-out is observed, which is good indication of poor adhesion between the filler and continuous polymer matrix.

Overall, the hybrid composites exhibit moderate to good filler distribution and dispersion. Interfacial adhesion can be improved in the processing of the composites such as double extrusion instead of hand-mixing of glass fiber before the injection molding step. Melt Blending (extrusion) is known to be an effective dispersive technique to incorporate fillers and it is expected that a second extrusion step would aid in greater dispersion of the filler. However, this would add costs to the overall part and the mechanical and thermal properties showed that the current methodology is good enough to be employed on non-structural components in body interior and underhood applications.

Summary and Recommendations

In this study, the development of hybrid composites were investigated utilizing DuPont's Nuvolve™ and glass fiber as fillers in a PP polymer matrix . The maximum total filler content did not exceed past 30 wt. % and the mechanical, thermal and morphological properties were evaluated and compared to Ford's incumbent material, neat PP and 5 %10% glass fiber filled composites for body interior and underhood applications in passenger vehicles and light-duty trucks.

The mechanical properties of the composites showed general increase in performance with glass fiber content and the opposite with Nuvolve™ content. However, optimum formulations were found (10/15, 10/20) that may reduce or replace a portion of the inorganic content. The best performing composites were 10/15 and 10/20 which outperformed Ford's incumbent material; creating an opportunity for a reduction up to 10% in total filler content and up to 13% in density per automotive part.

The thermal stability of the composites showed improvements in comparison to neat PP, where glass fiber had a more dominant role than Nuvolve™. The crystallization temperature (Tc) of the composites decreased revealing that Nuvolve™ is not a nucleating agent that could speed up crystallization during processing and subsequently decreasing cycle time. Overall, the composites showed improved thermal stability and can potentially be utilized in challenging environments such as underhood applications.

Morphology takes a deep dive into filler-matrix interactions which are pivotal for performance of the composites. DuPont's Nuvolve™ is a polysaccharide that is hydrophilic by nature and the PP is hydrophobic which highlights incompatibility issues. Therefore, having good distribution and adhesion of fillers are crucial to attain superior performance properties. The SEM micrographs showed that the best performing composites in this study (10/15 &10/20) had good to moderate filler distribution and adhesion with the continuous polymer matrix. There are localized areas with dark shadows around glass fiber and Nuvolve™ particles along with fiber pull-out showing glimpses of poor adhesion. This can be improved by adding a greater concentration of maleic anhydride or the addition of a second melt blending step.

The hybridization of Nuvolve™ with glass fiber yields high performing composites that can be exploited in body interior and underhood applications for passenger vehicles and light-duty trucks. The composites use up to 10% less filler material and demonstrate a density reduction of up to 13%; contributing to weight savings and also positively impacting the sustainability effort by incorporating a biodegradable material.

Acknowledgements

The co-authors and I would like to thank the Sustainability and Emerging materials group at Ford Motor Company that contributed to this work especially Daniel Frantz and Sandeep Tamrakar. We would also like to thank DuPont Industrial Biosciences for suppling raw material and the University Of Waterloo & Ford Motor Company for sponsoring this study.

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