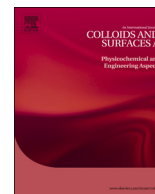




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Bicomponent poly(ethylene)/poly(propylene) fiber bonding using dielectric inks

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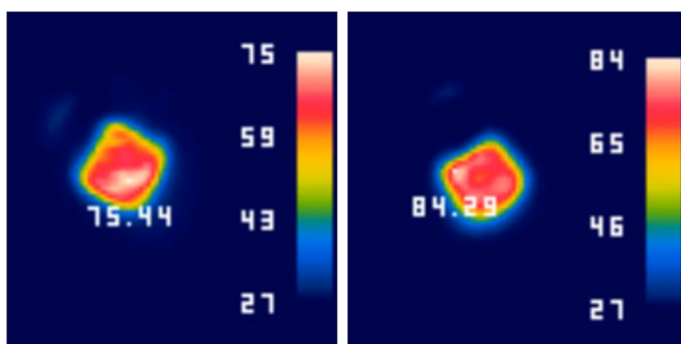
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GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

Fiber bonding
Microwave
Dielectric ink
Poly(ethylene)
Poly(propylene)

ABSTRACT

A dielectric ink thermal processing method has been developed to bond bicomponent poly(ethylene)/poly(propylene) (bico-PE/PP) fibers that are commonly used to make nonwoven fabrics for a variety of applications including medical, hygiene, and filtration needs. Dielectric inks applied to the fibers offer a number of potential advantages over conventional calendar bonding including more efficient energy conversion, faster heating rates, heating directed to desired bond sites, facile patterning, the ability to function on materials with substantial amounts of loft, and avoidance of fiber damage induced by calendar roller contact. Poly(ethylene) and poly(propylene) do not efficiently convert microwave energy to heat on their own; therefore, propylene carbonate (PC) and glycerine carbonate (GC) were selected as ink candidates due to their large dipole moments and dielectric constants. The temperature-dependent dielectric constant and dielectric loss values of both carbonates were measured as well as their thermal responses to microwave energy at 2.45 GHz. The dispersion of each carbonate on bico-PE/PP fibers, including the application to nonwoven fabrics, was characterized by optical microscopy. The thermal response for ink loading as a function of microwave radiation exposure time was

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<https://doi.org/10.1016/j.colsurfa.2019.123868>

Received 25 June 2019; Received in revised form 20 August 2019; Accepted 26 August 2019

Available online 27 August 2019

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investigated. Tensile test results showed that the bonding strength of dielectric ink bonded samples was comparable with the bonding obtained using conventional calendar bonding. The results presented herein demonstrate that dielectric inks provide fast heating rates, excellent fiber bonding, and the ability for facile nonwoven patterning.

1. Introduction

Microwave heating exhibits numerous potential advantages over conventional heating methods, namely: superior ramp rates, highly penetrative energy transfer via electromagnetic radiation rather than conduction, and improved heating selectivity and automation [1–3]. For these reasons, microwave heating has been used as an alternative to conventional heating both domestically and industrially for improved food cooking [4], rubber vulcanization [5], wood drying [6], sterilization of bacteria [7,8], and bonding of polymeric materials [9–11]. The focus herein addresses a key gap in the application of microwave to bond the industrially important class of bicomponent poly(ethylene)/poly(propylene) (bico-PE/PP) fibers widely used in the production of nonwoven materials. For this application, the use of microwave heating may be limited because poly(ethylene) and poly(propylene) have low susceptibility and are therefore largely unaffected by microwave radiation [1–4,12]. Therefore, strategies are needed to enhance the microwave response and improve the ability to convert electromagnetic energy into heat at bond sites. Enhancement methods published to date generally focus on doping target materials with nanoscale microwave receptors [1]. A broad range of nanostructured microwave receptors including ceramic materials, clay, carbon materials and dielectric polymeric materials have been reported [1,13–19]. However, with this method, heating rates of the doped materials are heavily dependent on the amount of receptor loading, which is consequently restricted by the base polymer. Additionally, receptors can have adverse effects on appearances and mechanical properties of the host polymers. Dielectric coatings including poly(aniline), poly(pyrrole) nanogranules, graphite, carbon black, metal, and metal oxide powders have been applied to poly(ethylene) and poly(propylene) to enhance microwave welding of bulk slabs of material [10,14,15,17,20–22]. Herein, we present the design and demonstration of a fast and industrially pertinent method to bond nonwoven fabrics consisting of micron-scale bicomponent poly(ethylene)/poly(propylene) (bico-PE/PP) fiber using microwave inks. This application of microwave inks is readily generalizable to nonwoven fabrics made from a variety of other thermoplastics.

Nonwoven fabrics are fabric-like materials made of a network of either staple or continuous fibers [23–25]. They have a wide array of applications in the medical field including use in gowns, scrubs, shoe covers and wound dressings. They are also widely used for making filters in everything from vacuum bags, to tea bags, to HEPA filters. Typically they are bonded together either through a mechanical, chemical, solvent, or heat treatment [26–31]. A common heat treatment method is calendar-bonding nonwovens by feeding them through heated rollers, heating, and thus bonding the materials by conduction. Due to the fact that the rollers are heated around their entire circumference, but only ever contact the fibers at one specific point, this method is inefficient for heat transfer. Calendar bonding can also damage and/or contaminate the fabric and new rollers are best employed for any changes in patterning desired. These shortcomings provide impetus for exploring a dielectric ink-based approach to microwave heating.

Dielectric ink applied to nonwoven fabric allows microwave heating without changing the structure and properties of microwave inactive fibers. Previous studies have reported the development of carbon nanotube and graphene containing inks [32–35]; however, the cost of these materials is prohibitive for large scale processing. In this study, readily available and cheap propylene carbonate (PC) and glycerine carbonate (GC) inks were employed (Fig. 1). These materials are

colorless, odorless liquids with low toxicity and high water solubility. The dipole moments of PC (5.36 D) and GC (5.05 D) suggest that they should be active under microwave radiation [36,37], and the boiling points of these two carbonates (PC 242 °C, GC 354 °C) are above the melting point of linear low density polyethylene (LLDPE). Based upon these physical properties, as well as viscosity and coating properties on the nonwoven fabrics, they were selected as the dielectric ink candidates for this study.

The PC and GC dielectric ink coating of nonwoven bico-PE/PP fabric results in preferential ink localization at fiber junctions that results in rapid, efficient, and localized heating under microwave irradiation. The LLDPE shell melts and bonds the fibers, while retaining the mechanical properties of the higher melting point PP core. This ink-assisted microwave thermal treatment can be applied to premade microwave inactive polymeric materials and can also be employed to create bonding patterns. The ink can be removed after successful bonding, thus retaining the original properties of target bonding materials.

2. Experimental

2.1. Materials

Propylene carbonate (PC) (ReagentPlus, 99%) and glycerine carbonate (GC) (90.7%) were purchased from Sigma-Aldrich (St Louis, MO) and used as received. Methanol (MeOH, HPLC grade) was purchased from Fisher Scientific (Hampton, NH). Ethanol (EtOH, Anhydrous) was purchased from Decon Laboratories, Inc (King of Prussia, PA). Commercial unbonded nonwoven fabrics composed of bico-PE/PP fibers had polymer densities of 0.955 g/cm³ and 0.900 g/cm³ for PE and PP respectively. Fibers had a 50:50 wt ratio of PE and PP and diameters of 20 μm resulting in an inner PP radius of about 7 μm and an outer LLDPE shell thickness of about 3 μm. On a mass per area basis, these nonwovens were 25 g/m². Commercial calendar bonded samples obtained for comparison had a mass per area of approximately 35 g/m². The bico-PE/PP fibers in the nonwoven fabrics consisted of Exxon Mobil PP Polypropylene Resin (Exxon PP3155) and Aspen 6850A Fiber Grade polyethylene (The Dow Chemical Company).

2.2. Methods

2.2.1. Dielectric measurement of propylene carbonate (PC) and glycerine carbonate (GC)

The dielectric constant (ϵ') and dielectric loss (ϵ'') values of PC and GC were measured at 20, 50, 80, and 110 °C using an N1501A Dielectric High Temperature Probe Kit (10 MHz–20 GHz) and an N9918A 26.5 GHz FieldFox Microwave Analyzer (200 MHz–26.5 GHz) (Keysight Technologies, Santa Rosa, CA, USA). Temperature control was achieved by placing a 10 mL sample within a 50 mL glass beaker

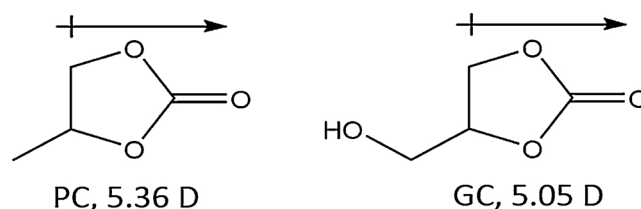


Fig. 1. Chemical structures and dipole moments of PC and GC.

that was partially immersed in an oil bath. Both samples were measured in triplicate.

2.2.2. Bulk microwave heating of PC and GC

1.00 ± 0.05 g samples of PC and GC were prepared in phenolic vial caps (13 mm height, 7 mm radius). Samples were rotated at 1.3 rps on the hotspot as they were heated at 1250 W in a modified Panasonic NN-SD762S Stainless 1250 W 1.6 Cu. Ft. Built-In/Countertop Microwave Oven with Inverter Technology (2.45 GHz) (Amazon.com, Seattle, WA, USA). Microwave exposure times reported refer to magnetron on time. For this oven, the magnetron turns on 4 s after the microwave timer starts. Bulk PC and GC samples were heated for 11 s (magnetron on time; 15 s total on microwave timer). This lag occurred in all microwave heating runs. Both samples were measured in triplicate.

2.2.3. Dispersion of ink onto bico-PE/PP nonwoven fabrics

Dielectric ink loading onto nonwoven bico-PE/PP fiber samples was determined by weighing before and after ink application. All sample coatings are reported in terms of grams ink per meter squared for fabric (g/m²). The samples were prepared using 10 wt% carbonate solutions dissolved in MeOH as this improved mass control of deposition on the fibers. 1 wt% red, glycerine-based food dye was also added to these inks to visualize distribution under an optical microscope. Ink was deposited onto the fibers using a 32 oz 'Nice! On The Move Clear Spray Bottle' (Walgreens Pharmacy, Deerfield, IL, USA). Once sprayed with ink within a chemical fume hood, the fibers were placed on a 70 mm Buchner funnel connected to a PIAB Lab-Vac H40 vacuum pump (-100 mbar) (Thermo Fisher Scientific, Waltham, MA, USA) to avoid the "coffee-ring" effect [38] and improve uniformity of ink distribution on the fabric. Optical images were captured using a Leica DMLP polarization microscope (Leica Microsystems GmbH, Wetzlar, Germany) equipped with an N Plan 5X objective and a SPOT Flex Mosaic Camera (SPOT 5.2 software).

2.2.4. Microwave heating of dielectric ink treated bico-PE/PP nonwoven fabrics

2 cm x 2 cm unbonded bico-PE/PP nonwoven fabrics were coated as described above with 10 wt% PC-MeOH or 10 wt% GC-MeOH ink. After MeOH evaporated, the samples were rotated at 1.3 rps as they were heated at high power (1250 W) for 5–10 seconds (1~6 s radiation).

2.2.5. D images of fabrics bonded by different approaches

3D images of fabric samples were captured using the Mid-Range x200 magnification of a MXB-2500REZ Digital Microscope (Hirox-USA, Inc., Hackensack, NJ, USA).

2.2.6. Tensile tests of GC-bonded nonwoven bico-PE/PP nonwoven fabrics

Tensile tests were conducted on 2 cm x 2 cm bico-PE/PP nonwoven fabrics bonded by the traditional heat processing method (calendar bonding) and GC-assisted microwave thermal processing. A 5940 Series Single Column Table Top Systems for Low-Force Mechanical Testing from Instron (Norwood, MA, USA) was used for the tensile tests. Samples were clamped to the test fixture along the fiber alignment with the 2 cm length and 1.5 cm width area pulled in the direction against the fiber alignment at a constant rate of 1 mm/s.

2.2.7. Ink patterning

A stencil was created by hollowing out an "M" on a piece of paper. 80 wt% GC/MeOH ink was prepared to minimize ink spreading on the non-woven surface. With the stencil positioned 1 cm from the fabric, ink was sprayed onto the sample. After allowing to dry, the sample was heated on the rotatable disc (1.3 rps) in the microwave oven for 5 s (1 s radiation).

3. Results and discussion

3.1. Temperature-dependent dielectric properties of PC and GC

Dielectric properties are temperature dependent parameters used to evaluate the heat storage and dissipation of materials. The relative permittivity or dielectric constant (ϵ') corresponds to how much electromagnetic energy can be absorbed by the material, while dielectric loss (ϵ'') indicates how efficiently the material converts the absorbed energy into heat. A high loss tangent ($\delta = \epsilon''/\epsilon'$) value generally indicates that the material will effectively convert microwave irradiation to heat [39,40]. Therefore, dielectric properties of pure PC and GC were measured at various temperatures to determine whether they were good candidates as dielectric inks for targeted thermal treatment applications.

At 2.45 GHz, the PC dielectric constant (ϵ') varied between 46 and 52 (~10% change) whereas the dielectric constant of GC increased greatly from 10 at 20 °C to 47 at 110 °C (~300% change). The PC dielectric loss value (ϵ'') was temperature dependent decreasing from 29 to 12 over the 20 °C to 110 °C temperature range whereas the value for GC increase from 14 to 32. Overall, these trends result in a substantially larger value of the loss tangent ($\delta = \epsilon''/\epsilon'$) for GC, in the range of 0.7–1.5, at all temperatures of interest (Fig. 2). Based upon the dielectric parameters, GC is expected to outperform PC in terms of achievable heating rates.

3.2. Heating performance of pure PC and GC

Bulk microwave heating of 1.00 g samples of PC and GC was performed to compare relative heating rates (Fig. 3). Within 2 s, the GC sample reached 120 °C, (above the 115–118 °C melting range of the LLDPE employed). Heating for times longer than 2 s resulted in curves that deviated substantially from linearity due to heat dissipation effects (Full details are provided in SI).

GC exhibited an average heating rate of 50 °C/s over 2 s whereas PC exhibited an average rate of 25 °C/s. As predicted from the dielectric data (Fig. 2), GC is a better candidate for microwave bonding if parameters such as mass of ink and/or rate of heating are important processing constraints. Additionally, the flash point of GC is above 190 °C, whereas the flash point of PC is 132 °C, which is close to literature melting range for LLDPE of 110–130 °C. Thus, from both an efficiency and safety standpoint, GC was selected as the dielectric ink for our studies of bico-PE/PP fiber nonwoven fabric bonding.

3.3. Dielectric properties and heating performance of GC/LLDPE composites

Microwave heating of poly(ethylene), and therefore bonding, can also be achieved by the formation of polymer composites. In order to directly compare the dielectric inks and polymer composite strategies, we prepared GC/LLDPE composites containing up to 10 wt% GC loading (larger amounts caused too great a change in other physical properties of interest). The dielectric constant and loss values of pure LLDPE and GC/LLDPE were compared under various temperatures to evaluate the heating performance of GC/LLDPE composites. Overall, the dielectric parameters of GC/LLDPE composites did not present significant improvement over pure LLDPE, which implied that GC/LLDPE would not heat efficiently. Microwave heating profiles of GC/LLDPE pucks confirmed this conclusion. Even for composites containing 10 wt% GC/LLDPE heating rates were only 2 °C/s, much lower than the ~50 °C/s GC heating rate (Full details are provided in SI). These results stand in contrast to those using conductive particles, such as metals and carbons, which are able to create a conductive, percolative network in a composite and generate a large microwave response [1,14,17,18].

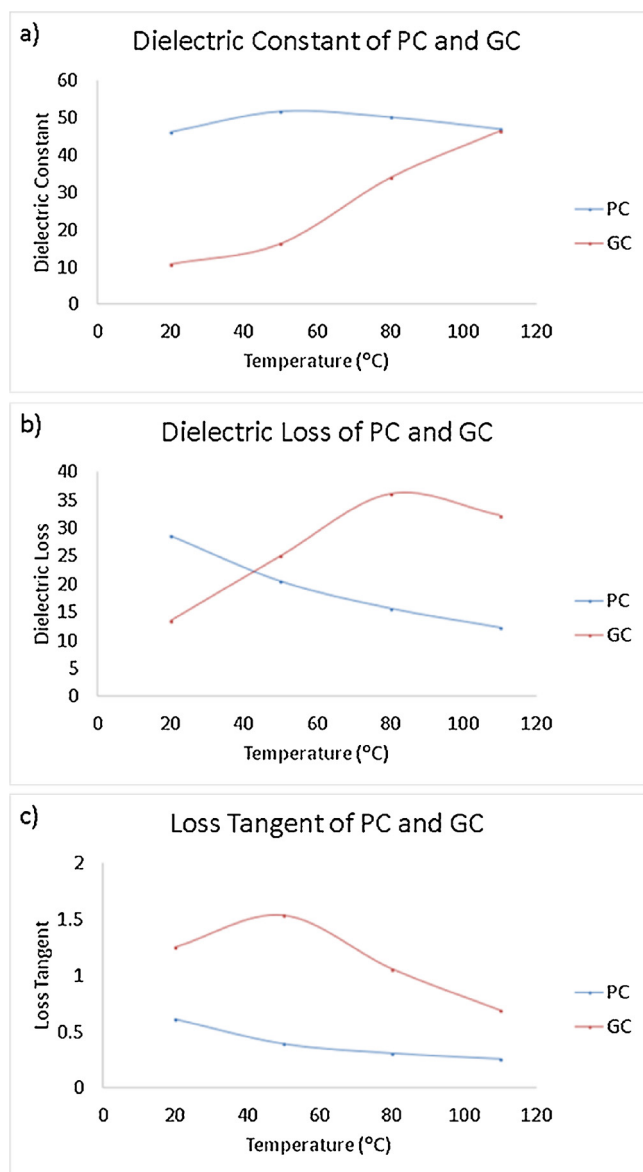


Fig. 2. The values of a) dielectric constant, b) dielectric loss and c) loss tangent of PC and GC at 2.45 GHz from 20 °C to 110 °C. Both samples were measured in triplicate with the average value reported at each temperature. Standard error bars are smaller than data points.

3.4. GC-MeOH ink dispersion on nonwoven fabrics

Optical microscopy was used to visualize GC distribution on the bico-PE/PP nonwoven fabrics. A 10 wt% GC/MeOH ink solution was sprayed onto the fabric to obtain a 50 g/m² coverage of GC. Fig. 4 illustrates the distribution of GC on the nonwoven fabric. An untreated nonwoven sample is provided for comparison. The GC is observed to agglomerate in areas with dense fiber crossing, as expected due to liquid surface tension. This distribution of ink has the potential benefit of localizing heat in the optimal locations to melt and bond fibers. No observable difference in GC dispersion on fabrics was detected after the microwave heating processing (SI).

3.5. Microwave treatments on nonwoven fabrics coated with GC-MeOH ink

Microwave thermal treatments were conducted on 10 wt% GC-MeOH coated bico-PE/PP nonwoven fabrics to investigate the ink loading threshold required to achieve effective bonding levels as

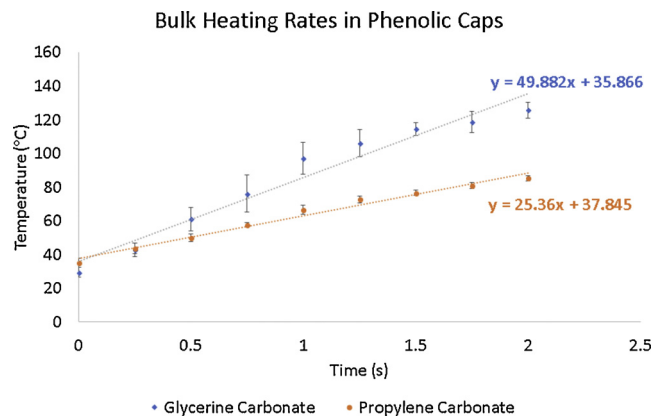


Fig. 3. Microwave heating rates of 1.00 g samples of PC (orange circles) and GC (blue diamonds). Both samples measured in triplicate (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

defined by mechanical testing. The preliminary, qualitative test of bonding employed was gently pulling the fibers to assess if they separated. If resistance was felt and samples did not immediately separate upon manual pulling, they were examined in the direction perpendicular to fibril alignment to determine bonding strength using quantitative tensile testing.

Samples coated with GC/MeOH were heated immediately after methanol evaporation. GC loadings and microwave radiation times are summarized in Fig. 5. Samples with the lowest ink loadings and/or shortest heating times failed to bond and fall in the region of the left of the band where good, functional fiber bonding was achieved. Samples containing greater ink loadings and/or longer heating times could result in burning and or bulk melting of the sample and fall in the region to the right of the effective bonding band. This data demonstrates that satisfactory bonding can be achieved within 1–5 s. The rapid heating rates achieved with GC ink are critical for practical processing conditions given the rate of nonwoven production and are in a similar range to results previously reported with HCl-doped polyanilines [9,20,21].

At 70 g/m² GC loading, the IR camera indicated a temperature of 90 °C reading after 1 s of microwave irradiation, which produced a completely bonded sample. Bonding was also achieved at lower 25 g/m² GC loadings as well; however, these samples plateaued at readings of 80 °C to 90 °C and required 3 s heating. The average microwave heating profile as measured by the thermal camera for 18 successfully bonded samples is shown in Fig. 6 (a thermal video is provided for a 47.5 g/m² loading). An average heating rate of 40 °C/s was observed in the first 1.5 s. Afterward the heating profile deviated substantially from linearity due to heat dissipation effects. A uniform ink dispersion (macroscopically) helped to decrease the ink loading threshold required for bonding purpose and allowed better control of the heating process.

Many GC samples that bonded had measured plateau temperatures between 80 °C and 90 °C, which is lower than the melting point of LLDPE. This effect is attributed to the resolution/depth of field of the IR camera utilized to measure the heating process. The temperature readings measured by the IR camera are a spatial average that includes the dielectric ink, the LLDPE shell, and the PP core. The camera does not resolve the temperature at the PE surface where bonding takes place. Therefore, the thermal camera is useful as a qualitative guide but not as quantitative measure.

3.6. Topology of fabrics bonded by different approaches

A comparison of unbonded nonwoven fibers, calendar-bonded nonwoven fibers, and GC ink/microwave bonded nonwoven fibers is provided in Fig. 7. Panel B highlights, as observable ovals, an example

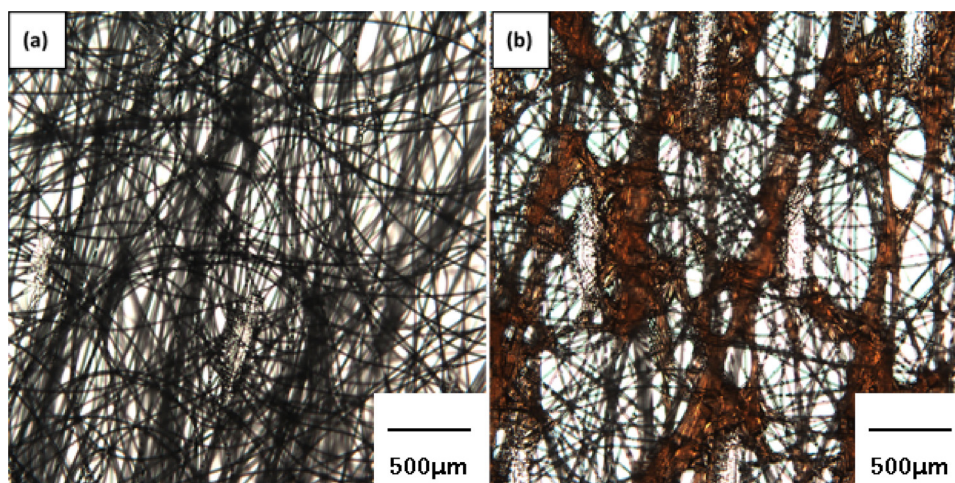


Fig. 4. Optical micrographs at 5X magnification taken of (a) untreated unbonded nonwoven fabric and (b) unbonded nonwoven fabric coated with 50 g/m² GC.

of fiber deformations occurring as a result of interactions with discrete calendar nips where maximum heat transfer occurs and maximum pressure is applied. Panel C presents a structure similar to Panel A, demonstrating that a key advantage of ink bonding method is to maintain the fiber topology of the original unbonded fabrics. This property of the GC ink is in contrast to the use of graphite powders, which resulted in melting of PP, incorporation of the powder into the plastic resulting in increased conductivity, and deformation of the PP around the graphite powder [14].

3D images of the surface of pre-bonded and post-bonded nonwoven fabrics have been compared. No significant difference in fiber alignments on the sample surface has been detected post-bonding (Fig. 8). The thickness of the unbonded fabric is ~270 μm whereas the after bonding that value decreases to ~210 μm. This change appears to be driven by surface tension effects of the GC ink. Other than the observed ~20% thickness compression, the GC-microwave bonded sample maintains the original nonwoven fiber structure, which is a key advantage of this novel ink-assisted thermal processing technology.

3.7. Tensile tests of GC bonded Bico-PE/PP nonwoven fabrics

Tensile tests of GC-bonded samples were performed to compare fabric yield strength to that of calendar-bonded fabrics. In this study, ultimate tensile stress (UTS) is the measured maximum stress (force per unit area) when the fabric starts to yield and tear, and is associated with

Average Heating Profile of GC Coated Fabrics

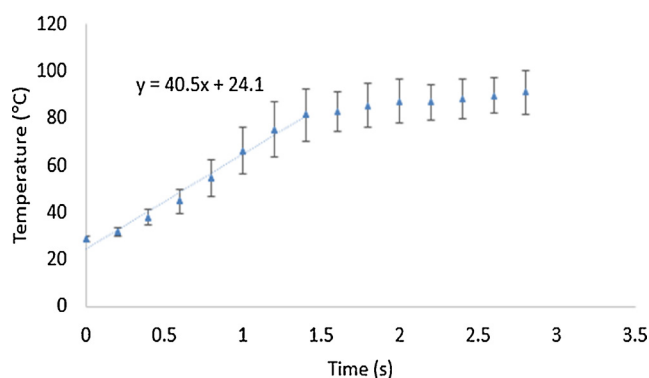


Fig. 6. Average microwave heating profile of 18 bico-PE/PP nonwoven samples coated with GC at a loading range of 25–70 g/m².

the highest point on the stress-strain curve (Table 1).

Tensile test results indicate that GC ink/microwave bonding provides equivalent bond strength (UTS of 20–30 KPa over range of conditions shown in Fig. 5) to that achieved by conventional calendar bonding. Samples bonded by different heating methods had equivalent Young's moduli, indicating that the ink heating method did not lower the mechanical properties of the fabrics. Therefore, from a mechanical

Heating Time vs Loading of GC-Bonded Bico-PE/PP Fabrics

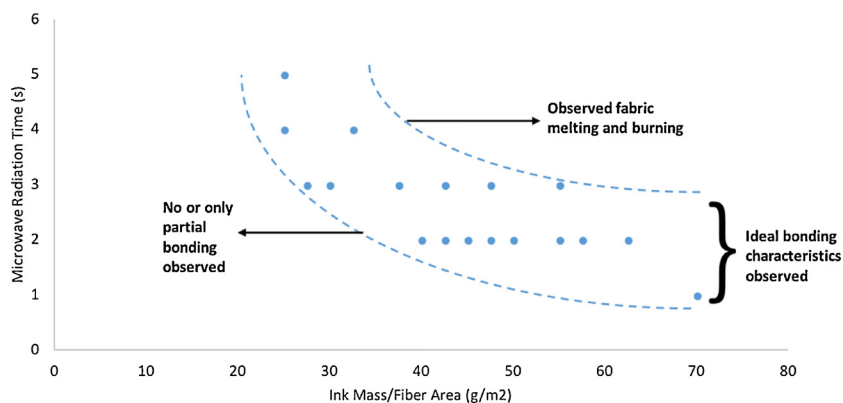


Fig. 5. GC loading versus microwave radiation exposure time for 2 cm x 2 cm bico-PE/PP nonwoven fabrics. Each data point indicates a particular ink loading/microwave exposure time coupled to a tensile test of bonding. The region in between the dotted lines highlights conditions that result in successful bonding.

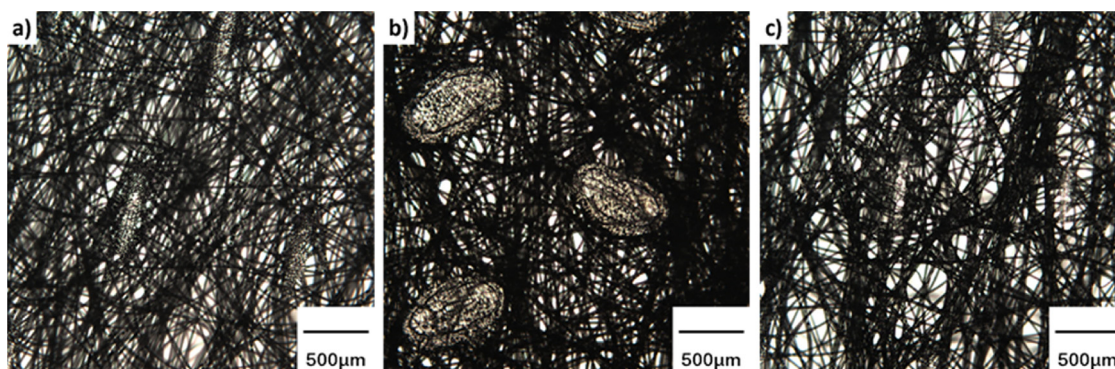


Fig. 7. Optical images of (a) unbonded, (b) calendar-bonded, and (c) GC-microwave bonded (GC washed off with MeOH) nonwoven fabrics.

property standpoint, GC ink bonding is a viable option to calendar bonding.

3.8. Ink patterning

GC ink can be utilized for heating the nonwoven in a desired pattern. Combined with compatible printers or designed stencils, dielectric ink can be distributed onto specific areas on the fabrics to achieve selective heating. A paper stencil with a hollow 4.8 cm wide M letter was created for ink patterning. With the stencil covering above fabrics, M patterns were formed by spraying ink through the stencil. Samples were heated on the rotatable disc at the primary hotspot in the microwave oven. Within one second the M heated to over 100 °C (Fig. 9), demonstrating the feasibility of patterned heating and bonding.

4. Conclusion

Glycerine carbonate and propylene carbonate were explored as dielectric inks for novel thermal bonding of nonwoven bico-PE/PP fabric due to their large dipole moments and boiling points.

Table 1

Average tensile stress values and Young's modulus of unbonded samples (6 tests), GC bonded samples (18 tests) and conventionally bonded samples (7 tests).

Sample	Unbonded	GC Microwave Bonding	Conventional Bonding
UTS (KPa)	3.2 ± 0.5	21.0 ± 5.0	19.5 ± 4.6
Young's modulus (KPa)	37.9 ± 3.8	41.3 ± 28.0	41.8 ± 5.5

Demonstration of these materials as effective microwave inks should be broadly useful for polymers with similar melting points to PE and PP and provide a readily accessible alternative to carbon-based (graphene, nanotube, graphite) active ingredients in ink formulations. They offer numerous advantages including substantially lower cost, low toxicity, they do not stain the fibers or fabric black, avoid damage introduced by calendar rollers, and do not impart electrical conductivity to the sample. The higher measured loss tangent of GC indicated that it should be preferable for microwave heating. Experimental heating profiles

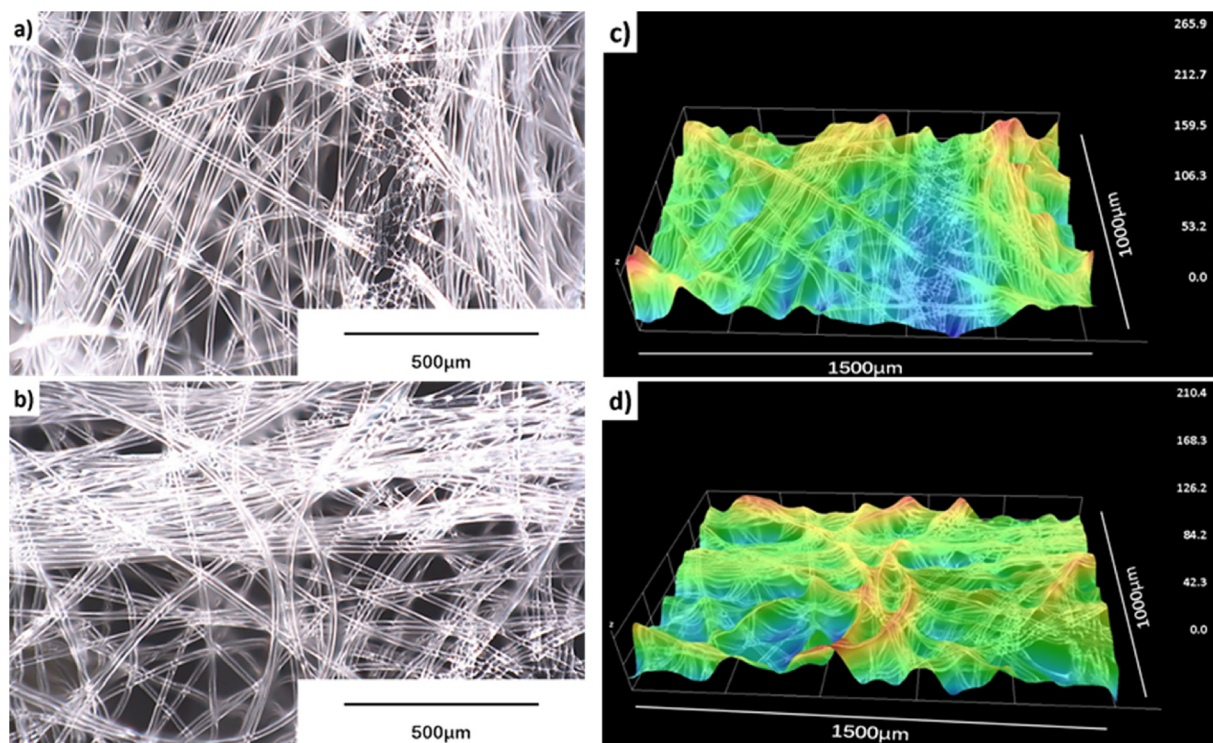


Fig. 8. Parfocal optical image of a) unbonded and b) GC-microwave bonded nonwoven fabrics and 3D optical image of c) unbonded and d) GC-microwave bonded nonwoven fabrics. The scale bars in a) and b) are 500 μm.

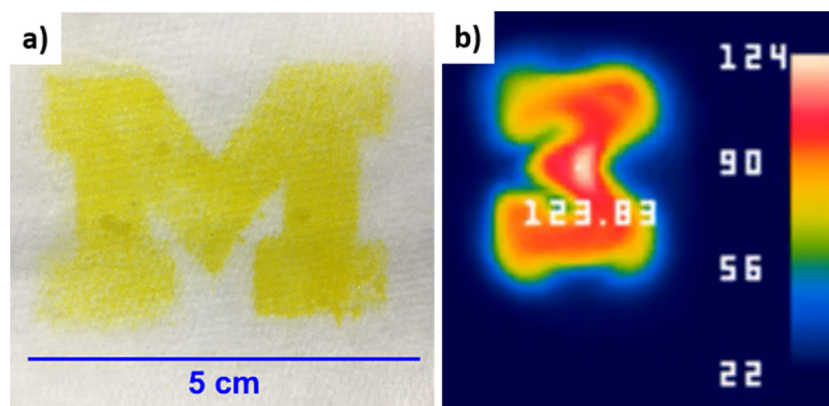


Fig. 9. a) GC ink pattern of M on bico-PE/PP fabrics and b) In situ IR image of sample after 1 s microwave radiation (1250 W).

confirmed that the heating rate of GC under microwave radiation at 2.45 GHz is approximately twice that of PC. This, in conjunction with the fact that PC's flashpoint is close to the melting point of polyethylene, made GC the preferred choice for further development. Optical images of GC coated on bico-PE/PP fibers demonstrated that GC tended to aggregate at fiber junctions, which further helps to focus the microwave energy to the appropriate location and avoid overall sample melting. Microwave treatments of GC coated bico-PE/PP nonwoven fabrics led to effective bonding as defined by mechanical testing. With GC loading between 25 g/m² and 70 g/m², bonding was achieved in 1–5 s. Fabrics bonded by both GC ink microwave treatment and the conventional thermal method (calendar-bonding) presented comparable tensile strength when pull-tested. Within the ink loading range for successful bonding, UTS was approximately equivalent regardless of whether samples were bonded at high ink loadings for short times, or low ink loadings for longer times. GC exhibits excellent potential for microwave-based thermal processing applications of premade materials to achieve fast and energy efficient heating. The work presented herein also provides guidance regarding the application and limitations of recent advances in thermal microscopy to evaluate polymer bonding. Although easy to implement and convenient for evaluation of patterned systems, the depth of field of the thermal analysis region limits quantitative assessment of temperature at the key surface contact points.

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.

Acknowledgments

This work was financially supported by a contract through the University of Michigan/Procter&Gamble Materials Innovation Collaboration. Adam J. Matzger is thanked for his advice and facilities for the optical micrographs.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.colsurfa.2019.123868>.

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