

# Processing Behavior, Morphology and Benefits of Using Low Density Hollow Glass Microspheres in Polymer Wood Composites

Steve E. Amos, Andy D'Souza, Baris Yalcin, Troy K. Ista, 3M Advanced Materials Division  
Craig M. Clemons, USDA Forest Service, Forest Products Laboratory

## Abstract

Low density hollow microspheres have been produced and used in various thermoset applications for decades. The advent of high-strength, low density glass microspheres that can survive high shear polymer processing operations such as injection molding in recent years is now allowing thermoplastics processors to successfully process these functional fillers with high survival rates, making target weight reductions possible.

In this paper, we will discuss the compounding methodology of hollow glass microspheres with high density polyethylene/wood flour in a twin screw extruder with subsequent die profiling and demonstrate processing and end use benefits. Morphology of the composites, mechanical and physical properties will be presented. The major benefits can be summarized as reduced density (lower than 1 g/cc) with zero to minimal sacrifice from moisture uptake and mechanical properties. Other end use benefits include sharper contours of the extruded profiles and improved nail-ability and screw-ability. Thermal behavior of the glass bubble/HDPE/wood composite profiles during processing and after the fact, i.e. thermal conductivity and diffusivity, will also be discussed.

## Introduction

Wood flour, in its as received form, is a low density filler (below 0.7 to 1 g/cc depending on type) that can be incorporated into low melting polyolefins such as polyethylene and polypropylene. One issue has been the increase in the density of the wood flour from below 1 g/cc to 1.4 g/cc when it is extruded or injection molded with polymers. This is due to compaction of the cellulosic walls by the compressive forces experienced during plastic processing. Increase in density of wood in processed plastics eliminates certain uses of these wood materials that require flotation in water. It also makes it very hard for the installer to nail and drill through the profiles. In order to attain real wood experience, decrease in the density of processed polymer wood composites has been exercised by foaming via chemical blowing agents. Foaming is known to decrease certain mechanical properties of wood such as its flexural strength. Water uptake in gas foamed polymer wood products has also been a challenge once water finds its path to the open pores. One alternative to foaming is the use of hollow glass microspheres.

## Experimental

### Materials:

Certene™ HGB-0760 High Density Polyethylene homopolymer in the form of pellets (Channel Prime Alliance) was the polymer used in the composites. STRUKTOL® TPW 104, a blend of lubricants designed specifically for wood fiber/flour filled polyolefins was used as the lubricant. Wood flour was Ponderosa Pine (Grade 4020) from American Wood Fibers. 3M™ Glass Bubbles iM30K (d: 0.6 g/cc with isostatic crush strength

of 28000 psi) and K42HS (d: 0.42 g/cc with isostatic crush strength of 7500 psi) (GBs) were used in extruded profiles.

### Processing:

The profiles were prepared in a two step process. First, a precompound formulation without any glass bubbles was prepared in a twin screw extruder. The wood flour and lubricant were dry blended and side stuffed while the HDPE was added through the main feed throat. The strands were pelletized into precompound-pellets. In the second stage, the precompound-pellets were introduced through the main feed throat while the glass bubbles were side stuffed into the extruder using the formulations in Table 1. The screw speed of the extruder was set to 50 rpm. The temperatures of the zones ranged from 182 to 171°C (360 to 340°F). Profiles were extruded at 12 in./min (18 lbs/hr) through a 2¼ in. x ¼ in. die, water cooled and dried at ambient temperature.

Formulation # Precompound (GB type)	WF Content (%)	Lub Content (%)	HDPE Content (%)	Glass Bubble Content (%)
1-A (iM30K)	50	6	44	0
2-A (iM30K)	47.5	5.7	41.8	5
3-A (iM30K)	45	5.4	39.6	10
4-A (iM30K)	42.5	5.1	37.4	15
5-B (iM30K)	40.7	4.9	35.9	18.5
6-A (iM30K)	45	5.4	39.6	10

Precompound	Wood Content (%)	Lub Content (%)	HDPE Content (%)	Glass Bubble Content (%)
A	50	6	44	0
B	47.4	5.7	46.9	0

Table 1. Formulations

## Characterization

### Density, glass bubble loading and survival rate

In order to determine the amount of glass bubbles compounded into the Wood/HDPE resin and the volume loss due to bubble breakage, the compounded pellets were exposed to high temperature in a Nabertherm® oven in order to volatilize the polymer resin and cellulosic fiber. The oven was set with a temperature ramp profile to run from 200 (392°F) to 550°C (1022°F) in 5 hours. After the temperature reached 550°C (1022°F), it was kept constant for 12 hours. The amount of inorganics, i.e. glass bubbles, was calculated from the known amounts of resin (wood/HDPE) before and after the burn process. In order to determine the amount of volume loss due to bubble breakage, the residual material after burn off, which is mainly glass bubbles, was calculated for its density using a helium gas pycnometer (AccuPyc™ 1330 from Micromeritics). The density values of wood composite samples (dry and wet) were also determined by the gas pycnometer.

### Morphology

SEM images were taken using a FEI® SEM system in order to assess glass bubble distribution.

### Thermal properties

Thermal conductivity K-value (W/mK) was measured using a LaserComp FOX50 thermal conductivity measurement system. For thermal conductivity, K-value samples were cut out from extruded profiles and mechanically planed in the form of a disk with 50 mm diameter and 6 mm thickness. In order to assess the effect of glass bubbles on the cooling rate from the melt, prefabricated profiles were heated to their partially molten stage in a compression molder followed by their quick removal to an ambient room temperature and temperature measurement using a non-contact IR camera.

### Mechanical

Flexural properties was measured on a Sintech 1/G system. A modified version of ASTM D790 three point bending test was used. Izod impact strength was determined using a Tinius Olsen Izod impact measurement system. The samples were prepared according to the ASTM D256 standard. It is important that the values (flexural modulus, flexural strength, izod impact strength) obtained using these instruments are not absolute values and should be compared relative to the samples in this study.

## Results and Discussion

The density of HDPE/wood profiles was successfully reduced to below 1.0 g/cc, allowing them to float in water (Figure 1). About 90% by volume of the glass bubbles survived in highly filled formulations (Table 2).

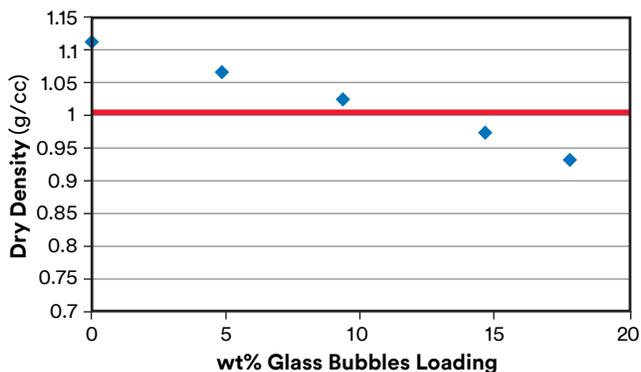


Figure 1. Density of HDPE/Wood composites as a function of GB loading

Sample ID	Theoretical Weight % Glass Bubble Loading %	Actual Weight % Residue (wood ash + GB) %	Theo. density of GB (g/cc)	Burn-off Residue Density (g/cc)	Volume Loss Due to Bubble Breakage (%)
1-A	0	0.63	n/A	n/A	n/A
2-A	5	5.51	0.6	0.7249	17.23%
3-A	10	9.94	0.6	0.6846	12.36%
4-A	15	15.26	0.6	0.6749	11.10%
5-A	18	18.46	0.6	0.6806	11.84%
6-A	10	10.38	0.42	0.6057	30.66%

Table 2. Glass Bubble Loading and Vol % survival rate

SEM images evidenced uniform dispersion and high survival rates of glass bubbles in WPCs (Figure 2). The presence of glass bubbles increased the flexural modulus while decreasing density (Figure 3). Impact properties are preserved 70% compared to the original value. Glass bubble filled profiles also exhibited increased cooling rates from the melt, allowing faster production rates (Figure 4). Glass bubble filled formulations also showed lower thermal conductivity (Figure 5).

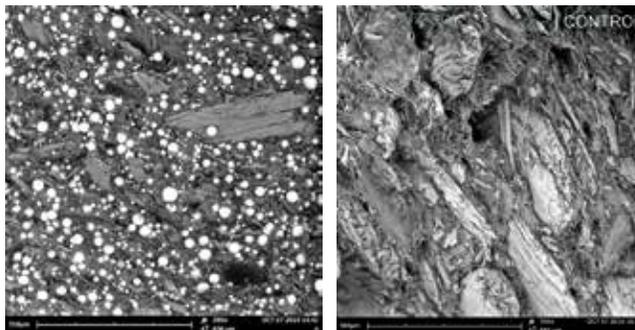


Figure 2. Scanning electron microscopy

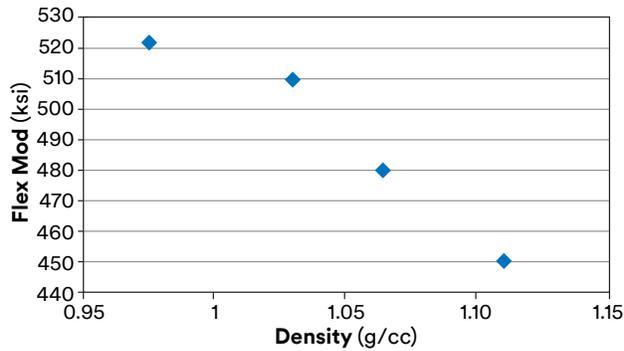


Figure 3. Flexural Modulus as a function of density

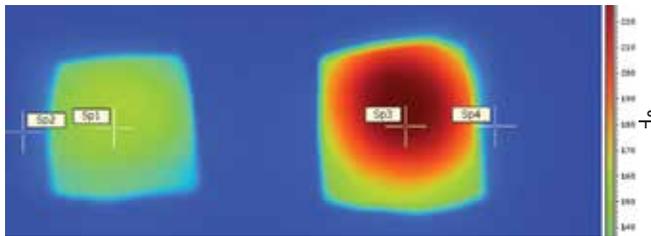
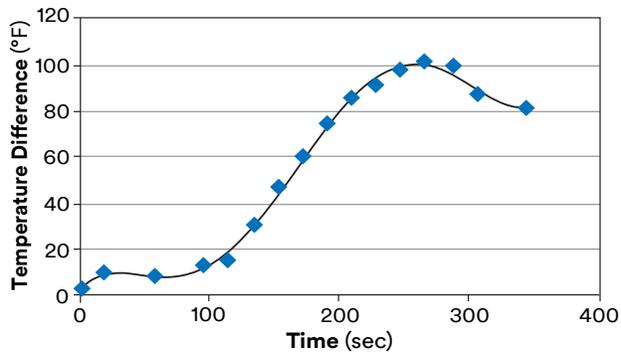
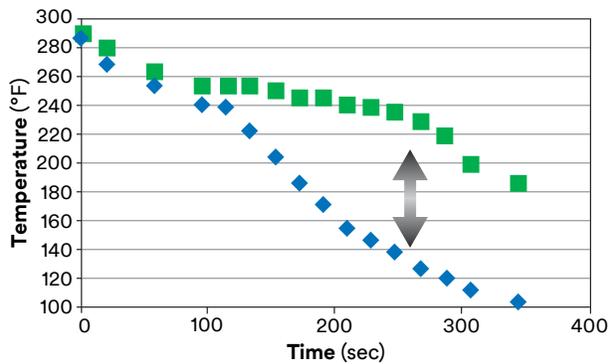


Figure 4. Cooling from the melt with (left) and without (right) glass bubbles



One of the drawbacks of standard wood/polymer composites is that it is harder to nail and screw into the composite material as compared to regular wood. This is mainly due to the presence of a dense polymer phase with limited void volume. With the incorporation of glass bubbles into the wood composite, the composite accepts screws and nails more like real wood than do their counterparts without glass bubbles. In order to demonstrate this effect, a simple experiment was performed. Two profiles, one with 15 wt% glass bubbles and the other without any glass bubbles, were drilled using a heavy duty drill with a ¼ in. drill bit. The electric drill was held on the wood composite profiles without applying any extra pushing force other than the weight of the drill. One can approximate the pressure at the wood-drill bit contact point by dividing the weight of the drill by the cross sectional area of the ¼ in. drill bit. 1800 g drill weight applies a pressure of ~80 psi at the contact point. With this pressure, it takes about 1 minute and 15 seconds for the drill to go through the ¼ in. thick profile without any glass bubbles, while it takes only 15 to 20 seconds for the profile with the glass bubbles. The glass bubbles, when broken due to contact with the drill bit, afford void volume for the drill bit to penetrate through. The same observation is true with nails. Nails can be inserted with much less effort into the composites with glass bubbles.

Glass bubbles also render sharper contours and corners in the extruded profiles as well as better surface definition than profiles without any glass bubbles.

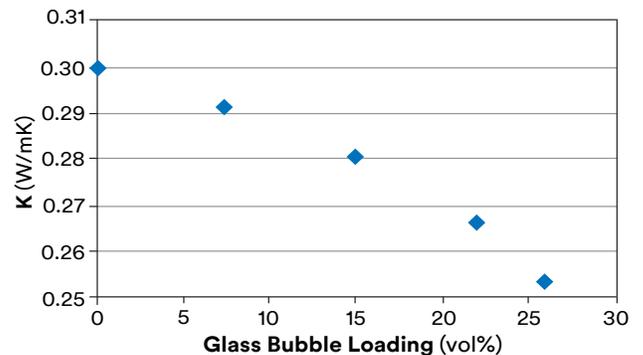


Figure 5. Thermal conductivity as a function of glass bubble loading

## Conclusions

Glass bubbles in polymer wood composites reduce density while increasing flexural modulus. They reduce thermal conductivity of wood, impart ease of nailing/drilling and render sharper contours. From a processing point of view, glass bubbles do not increase the melt viscosity despite increased filler loading. Extruder load decreases with increased glass bubble content. Increased cooling rates are achieved in glass bubble filled composite profiles that could result in faster production rates.

**Note:** The purpose of this paper is to provide basic information to product users for use in evaluating, processing, and troubleshooting their use of certain 3M products. The information provided is general or summary in nature and is offered to assist the user. The information is not intended to replace the user's careful consideration of the unique circumstances and conditions involved in its use and processing of 3M products. The user is responsible for determining whether this information is suitable and appropriate for the user's particular use and intended application. The user is solely responsible for evaluating third party intellectual property rights and for ensuring that user's use and intended application of 3M product does not violate any third party intellectual property rights.

**Warranty, Limited Remedy, and Disclaimer:** Many factors beyond 3M's control and uniquely within user's knowledge and control can affect the use and performance of a 3M product in a particular application. User is solely responsible for evaluating the 3M product and determining whether it is fit for a particular purpose and suitable for user's method of application. User is solely responsible for evaluating third party intellectual property rights and for ensuring that user's use of 3M product does not violate any third party intellectual property rights. Unless a different warranty is specifically stated in the applicable product literature or packaging insert, 3M warrants that each 3M product meets the applicable 3M product specification at the time 3M ships the product. 3M MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESS OR IMPLIED, INCLUDING, BUT NOT LIMITED TO, ANY IMPLIED WARRANTY OR CONDITION OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE OR ANY IMPLIED WARRANTY OF NON-INFRINGEMENT OR ANY IMPLIED WARRANTY OR CONDITION ARISING OUT OF A COURSE OF DEALING, CUSTOM OR USAGE OF TRADE. If the 3M product does not conform to this warranty, then the sole and exclusive remedy is, at 3M's option, replacement of the 3M product or refund of the purchase price.

**Limitation of Liability:** Except where prohibited by law, 3M will not be liable for any loss or damages arising from the 3M product, whether direct, indirect, special, incidental or consequential, regardless of the legal theory asserted, including warranty, contract, negligence or strict liability.

**Technical Information:** Technical information, recommendations, and other statements contained in this document or provided by 3M personnel are based on tests or experience that 3M believes are reliable, but the accuracy or completeness of such information is not guaranteed. Such information is intended for persons with knowledge and technical skills sufficient to assess and apply their own informed judgment to the information. No license under any 3M or third party intellectual property rights is granted or implied with this information.



**3M Advanced Materials Division**

3M Center  
St. Paul, MN 55144 USA

Phone 1-800-367-8905  
Web [www.3M.com/glassbubbles](http://www.3M.com/glassbubbles)

Please recycle. Printed in USA.  
©3M 2016. All rights reserved.  
Issued: 10/16 11835HB  
98-0212-4213-0

Certene is a trademark of Muehlstein.  
STRUKTOL is a registered trademark of Struktol. Nabertherm is a registered trademark of Nabertherm GmbH.  
AccuPyc is a trademark of Micromeritics Instrument Corporation. FEI is a registered trademark of FEI Company. 3M is a trademark of 3M. Used under license by 3M subsidiaries and affiliates.