

Improving the sintering behavior of mesocarbon-microbeads for the manufacture of high quality carbon products using a joint promoter comprising carbon black and glycidyl methacrylate



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HIGHLIGHTS

- Qualities of sintered MCMB product was effectively improved by CB and GMA additives.
- CB and GMA exert a mutual cooperative effect on the sintering reactions of MCMBs.
- High bending strength (142 MPa) carbonized carbon-blocks were obtained.

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ABSTRACT

Carbon-black (CB) and glycidyl methacrylate (GMA) were used together as a joint promoter to improve the quality of sintered mesocarbon products. The results show that, in addition to inhibiting the condensation of β -resin at $\sim 200^\circ\text{C}$, GMA reduces the formation of volatile pyrolysis products during MCMB sintering at $\sim 300\text{--}550^\circ\text{C}$, while CB accelerates pyrolysis and lowers the temperature at which the evolution of volatile components begins. Our observations suggest that a cooperative effect, between CB and GMA, improves sintering behavior during sintered mesocarbon product manufacture, resulting in the formation of carbonized carbon blocks with high bending strengths (142 MPa) and high densities (1.87 g cm^{-3}).

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1. Introduction

The manufacture of high performance carbon blocks is attracting significant attention, due to their increasing use in high-technology applications, e.g. in the electric, metallurgical, nuclear, and chemical industries. Mesocarbon-microbead (MCMB) sintering is recognized as leading manufacturing method because of the excellent compressibility, and the rapid low temperature sinterability of MCMB materials [1–6]. Although the sintered mesocarbon products have high densities and good friction characteristics, they have unacceptable mechanical properties [5–7]. Much effort has been devoted to studying MCMB sintering reactions and improving the mechanical properties of the resulting products. Various additives, such as: ZrB_4C [5], Ti/Ni [8], carbon-

black (CB) [9,10], carbon-nanotube [11], SiC [12], and Ti [13] have been shown to promote the sintering reaction and improve the products' mechanical strength. In addition to the kinetic aspects of the reaction, the physical properties of the viscous raw materials, e.g. their contacting patterns and fluidities, are critical determinants of MCMB sintering outcomes. A higher β -resin content (i.e. the difference between toluene insoluble (TI) and quinoline insoluble (QI) compounds) and a better MCMB contacting pattern were found to favor the fluidity and sinterability of the 'clumpy' mixtures and to improve the bending strength of the products [11,14–16]. The reason for this is that the reactions are not fully homogenous [14–17]. However, the β -resin content of the MCMB raw materials must be limited, as an excess will promote bursting of the carbon matrixes, due to the release of volatiles during degradation [15,18,19]. The fluidity change of the raw materials can also affect neck-formation [2] between the MCMBs and consequently alter the product's resulting strength. The viscosity of the raw materials decreases initially with an increase in temperature,

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but increases rapidly when the sintering temperature is higher than the onset temperature for β -resin condensation [20]. The viscosity increase (hardening of the materials) at the initial low temperature stage tends to hinder neck-formation among the MCMBs. Taking these facts into account, and remembering that the β -resin content is a key factor in MCMB sintering [4,6,21,22], it is apparent that promoting material fluidity and reducing the destructive effects from pyrolysis gas evolution are important strategies for improving the quality of sintered mesocarbon blocks. However, very few studies have focused on these aspects, thus techniques for producing high quality sintered mesocarbon products need further investigation.

In this work, CB and glycidyl methacrylate (GMA) were used as joint reaction-promoters to modify the sintering behavior of the β -resin and improve the quality of the sintered mesocarbon products. GMA provides reactive functionality (epoxy and methacrylate) [23,24] to react with the intermediate species from the pyrolysis reaction to reduce the formation of volatile gases, while CB accelerates pyrolysis during sintering [9,10]. The effects of the promoters on improving the sintering behavior of β -resin, and enhancing the mechanical strength of the sintered mesocarbon products, were investigated.

2. Experimental details

Solid-resin containing a high β -resin content (65.6 wt %) and a low QI content (12.4 wt %), and MCMBs having a high QI content (ca. 99 wt %) and a low β -resin content (0.3–0.6 wt %) were provided by the China Steel Chemical Corp. (Taiwan). The average particle sizes of the solid resin and MCMB were 4.87 and 10.74 μm , respectively. The MCMBs were completely blended with the solid-resin and the various additives (BP-2000 CB 8 wt %, GMA 2 wt %) using a high-speed mixer (HSM-25, She-Hui Machinery Co., Taiwan) in dry form at 200 rpm for 15 min, and then submitted for modeling by cold isotropic compression (1600 kg cm^{-2}) in a cylindrical rubber mold (7.70 cm diameter and 14.2 cm length) using a Nikkiso CL-49-22-30 cold isostatic presser. The β -resin content of the green sample blocks was altered by changing the solid-resin/MCMB ratio. The β -resin contents had the same composition. These qualities facilitated examination of the dependence of the sintering behavior and the resulting product quality on the reaction-promoters and β -resin contents. The green sample blocks were carbonized in a Carbolite CWF-1100 furnace using a carbonization time-temperature sequence given in our previous paper [15]. The weight of the samples, before and after the carbonization, was measured in milligrams to determine the weight loss during the procedure. The molecular weight distribution of the solid-resin was determined using a MALDI-TOFMS (Autoflex III, Bruker Daltonics GmbH, Leipzig, Germany). The thermal behavior of the solid-resin and the MCMB raw material was examined by thermogravimetry (TGA) using a Perkin-Elmer SDT-Q600 Thermogravimetric Analyzer/Differential Thermal Analyzer (DTA). The bending strength of the sintered mesocarbon blocks was measured using a three-point fixture on an MTS Sintech 10/GL testing machine (method based on ASTM C 1161-02), while the density was measured by the Archimedes drainage method. Scanning electron microscopy (SEM) images of the carbon blocks were obtained using a JEOL-JSM 6300 SEM.

3. Results and discussion

3.1. Effects of GMA and CB on the molecular weight distribution and β -resin content of the heated solid-resin

The solid-resin, with and without GMA and CB, was heated at 170, 190 or 210 $^{\circ}\text{C}$ in a nitrogen environment for 2 h to examine the

efficacy of the reaction-promoters in inhibiting material condensation during the initial low temperature sintering process. The results, see Fig. 1, show that the heated (190 and 210 $^{\circ}\text{C}$) original solid resin (SR) show additional higher molecular weight peaks that result from condensation polymerization of low molecular weight compounds into larger molecular species (e.g. 2300 g mol^{-1}) [20,25,26], while the heated samples with the GMA (SR_{GMA}), CB (SR_{CB}) and CB + GMA (SR_{CG}) do not.

These results reveal that condensation polymerization of the solid-resin at ~ 200 $^{\circ}\text{C}$ was inhibited by GMA and CB.

The TI, QI and β -resin contents of the various heated solid-resins (measurements based on ASTM D 7280 and JISK 2425), are shown in Table 1. The results indicate that the fraction of higher molecular weight components (determined from TI and QI values), of the heated SR, are higher than those of SR_{GMA}, SR_{CB} and SR_{CG}. For example, the SR heated at 190 $^{\circ}\text{C}$ has a higher TI value (95.5%) compared to SR_{GMA}, SR_{CB} and SR_{CG}, whose TI values are 87.4%, 88.1% and 86.6% respectively. These results and those in Fig. 1 clearly show that the degree of condensation of the solid-resin, occurring at ~ 200 $^{\circ}\text{C}$, was significantly reduced in the presence of GMA and CB. In addition, the results also show that the TI (89.2%) and QI (62.4%) contents of the heated SR_{CG} are lower than those of the

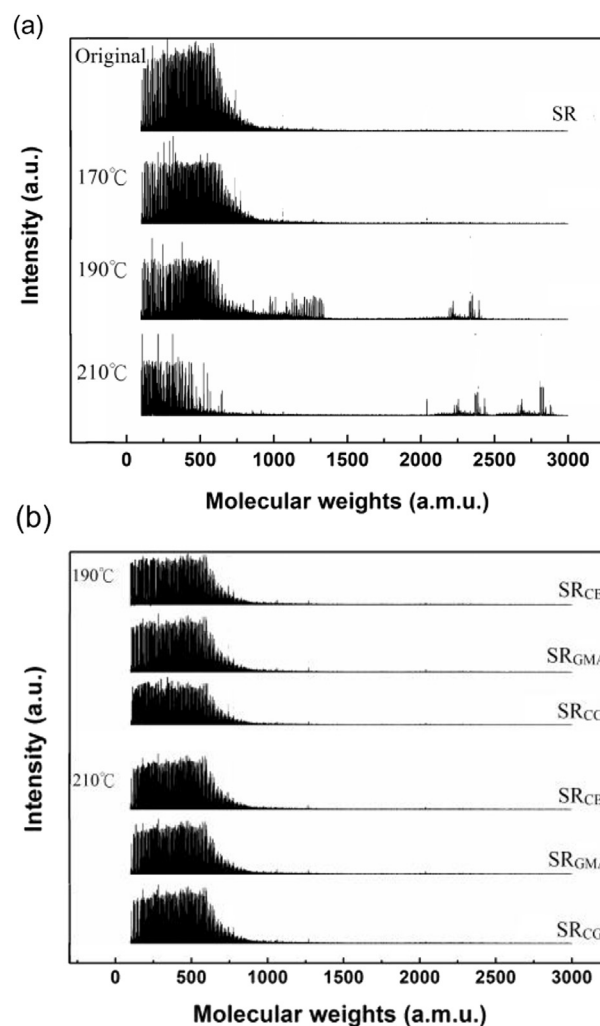


Fig. 1. Molecular weight distribution of: (a) the original and heated SR, (b) heated SR_{CB}, SR_{GMA}, and SR_{CG}. SR: solid-resin without additives. SR_{CB}, SR_{GMA}, and SR_{CG} are solid-resin with CB, GMA and CB + GMA, respectively. Additive content: CB 8 wt %, GMA 2 wt %.

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