Polymer xxx (2016) 1–11

Contents lists available at ScienceDirect

Polymer

journal homepage: www.elsevier.com/locate/polymer

# In-situ addition of graphene oxide for improving the thermal stability of superhydrophobic hybrid materials

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ARTICLE INFO

*Article history:*
Received 3 November 2016
Received in revised form
28 December 2016
Accepted 29 December 2016
Available online xxx

*Keywords:*
Superhydrophobic
Polymethylhydroxysiloxane
Cetyl trimethylammonium bromide
Graphene oxide
Chloroform detection and separation

ABSTRACT

Superhydrophobic mesoporous hybrid materials were synthesised by the in-situ self-hydroxylation and condensation of polymethylhydrosiloxane in ethanol and sodium hydroxide solutions in the presence of a cetyl trimethylammonium bromide (CTAB) as a surfactant and graphene oxide (GO). The samples were analysed by a range of characterisation techniques, such as Fourier-transform infrared and Raman spectroscopy, $^{29}$Si cross polarisation magic angle spinning nuclear magnetic resonance spectroscopy, X-ray diffraction, X-ray photoelectron spectroscopy, surface area analysis, high resolution scanning electron microscopy, and high resolution transmission electron microscopy. The superhydrophobic hybrid powder was used for the detection and separation of chloroform in water from water/chloroform mixtures.


## 1. Introduction

In the recent days, superhydrophobic materials have attracted considerable attention in environmental applications, such as sorption and the separation of various oils and organic solvents, selective removal of metal ion adsorption, and gas sensors [1–10]. In general, superhydrophobic surfaces can be prepared easily by enhancing the surface roughness or by reducing the surface energy of the materials using low surface energy materials [11,12]. Fluorine-based low surface energy materials are used widely to prepare superhydrophobic materials [13]. Fluorine-based compounds, however, are more expensive than other compounds, which has limited their use in some practical applications. Several polymers, silane precursors, metal ions, and various methods have been used for the synthesis and fabrication of superhydrophobic materials and surfaces [14–19].

Polymethylhydrosiloxane (PMHS) is a well-known siloxane material used in a range of applications, such as stable hydrophobic coatings, highly transparent substrates, reducing agents for organic synthesis, biocompatible materials, micro-pattern devices, and nano necklace fabrication [20–22]. Yang et al. synthesised a non-template superhydrophobic micro-mesoporous hybrid using PMHS and tetraethoxysilane (TEOS) and reported that more than one week was needed to reproduce the superhydrophobic hybrid micro/mesoporous material [23–25]. PMHS exhibited self-hydroxylation and condensation properties in sodium hydroxide solutions [11,23–25]. Therefore, superhydrophobic mesoporous material were synthesised in a one-pot process (within 2–3 days) using PMHS (without a cross-linking agent and surfactant). The superhydrophobic material was used for the preparation of novel superhydrophobic hybrid materials using natural leaf powder for oil spill capture and metal ions adsorption [11,26].

Graphene oxide (GO) is a well-known carbon material because of its excellent properties, such as easy dispersion in water and other organic solvents, electrical insulator, and readily modified by chemical or physical methods [27,28]. GO is anchored with covalently bonded functional groups, such as carbonyl or carboxyl, epoxy, and hydroxyl groups [29]. The above functional groups present in GO makes it a hydrophilic nature. The functional groups are active to react with various active molecules to form covalent or weak Van der Waals force of attraction. The addition of GO can enhance the thermal, mechanical, electrical, and other properties of the final materials [30,31]. GO is used widely in energy, environmental and biological applications due to the excellent properties [28].

In this study, cetyl trimethyl ammonium bromide (CTAB, ≥99%) was introduced as a surfactant and the solvent removal temperature was maintained at 150 °C for further condensation of the

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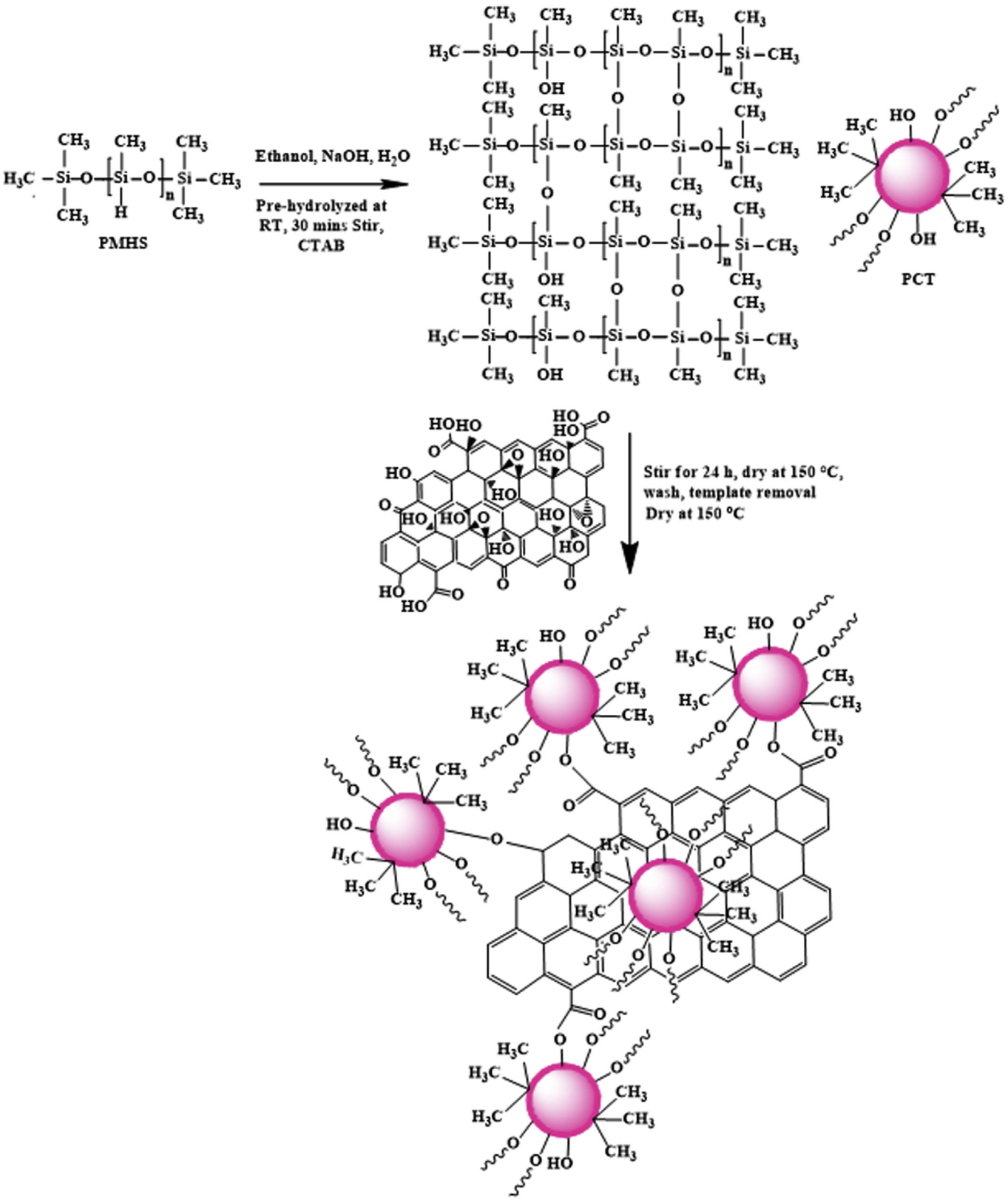
http://dx.doi.org/10.1016/j.polymer.2016.12.072
0032-3861/

hydrolysed siloxane network. The surfactant was used to ensure the porous structure of the synthesised material and was removed from the material using a solvent extraction method. Furthermore, the in-situ addition of graphene oxide (GO) to the pre-hydrolysed PMHS was also studied. This paper reports the results of a detailed study of the structural, thermal properties of the superhydrophobic hybrid materials made from PMHS and GO or PMHS in the presence of CTAB surfactant by various characterisation tools.




**Scheme 1.** Synthesis of superhydrophobic polysiloxane/GO hybrid material.

Please cite this article in press as: S. Nagappan, C.-S. Ha, In-situ addition of graphene oxide for improving the thermal stability of superhydrophobic hybrid materials, Polymer (2016), http://dx.doi.org/10.1016/j.polymer.2016.12.072