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## Synthesis and Characterization of Urea-formaldehyde Microcapsules Containing Functionalized Polydimethylsiloxanes

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### Abstract

Self-healing anticorrosive coatings require healing agents with hydrophobic properties and low glass transition temperature. Polydimethylsiloxanes are group of compounds with high degree of hydrophobicity and very low glass transition temperatures. The nature of functional group of core material highly affect the properties of resultant microcapsules. Therefore, in this work, three polydimethylsiloxanes with different functional groups were successfully encapsulated in urea-formaldehyde microcapsules for self-healing applications through oil-in-water (OIW) emulsion polymerization. The resultant microcapsules were analyzed via Fourier transformed Infrared spectroscopy (FT-IR) for structural confirmation, thermogravimetric analysis (TGA) for the thermal behaviour, scanning electron microscopy (SEM) for size and morphology, and electron dispersive spectroscopy (EDS) for the elemental composition. For OH and vinyl terminated PDMS, microcapsules had diameters in the range of 100 to 500  $\mu\text{m}$  while for Epoxy terminated PDMS microcapsules diameters were in the range of 50 to 100  $\mu\text{m}$ . Thermal degradation behaviour was also different for all kinds of microcapsules, showing that nature of core materials affect the properties of microcapsules.

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### 1. Introduction

During the last 10 to 15 years, the smart self-healing composite systems have been the major area of research for material scientists and researchers. The main goals and objectives are to design smart polymeric self-healing system

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with inborn ability of self-reparation, reinstating the physical and mechanical properties of the composite structural materials rapidly after being subjected to injury. The self-reparation property of materials is a unique and valued characteristic because it increases the chances of longer service and shelf lives of materials which in turn contribute towards the financial and human safety attributes. In thermosetting polymer applications, various kinds of healing triggering mechanisms have been proposed and virtually applied [1, 2].

Mechanical, thermal and photo-initiated self-healing mechanisms are the most commonly applied repairing techniques [3, 4]. Microencapsulated self-healing approach is the most common type of mechanical stimuli which is superior to other self-healing techniques owing to the simplicity, ease of microcapsules synthesis, robustness, and simple use in coatings systems. Generally speaking, microcapsules are small spheres in the range of micrometres encircling liquid materials (core material), with in various types of shell walls. Solids, liquids and gasses could be easily encapsulated inside these polymeric walls depending upon the required applications [5]. The synthesis and final structure of microcapsules perform a vital part in the performance of the self-healing coating. Preferably, microcapsules should be stable towards acids, bases and moisture, should pose non-leaky shell wall, should be able to contain the core materials for longer period of time, and only release the core contents whenever required [6]. Commonly, polymer and polymers composite systems are treated at elevated temperature and higher shear stresses. Epoxy group of resins and other thermosetting polymers are usually cured in the range of 100-200°C. Thermoplastic polymers are extruded above 150°C as well as at higher shear stresses. The requirements of polymers in such harsh environment necessitate further improvements in stability of microcapsules for applications in polymer composite systems [7]. A strong shell wall is one of the key factors that determine the robustness of microcapsules. However, some critical aspects like the chemical behaviour of the core material towards shell wall material, processing conditions of synthesis, viscosity of the healing agent and possible dissolution of shell polymer are the major deciding factors as well. The chemical and physical properties of shell wall material are very crucial. Unlike classes of healing chemistries cannot be encapsulated in alike shell wall or identical shell wall for dissimilar type of compounds do not perform in the same way [8]. The storage capabilities of microcapsules is an important aspect, yet no proper research has been conducted to find out the appropriateness of unlike core materials with different polymeric shell walls. This study aimed to find the suitability of urea-formaldehyde shell wall for different PDMS cores.

Although epoxy resins have very good properties for the protection of a number of materials, yet for corrosion prevention some hydrophobic part should be introduced into epoxy backbone. Polydimethylsiloxanes can be used for this purpose. The group of silicon containing compounds containing dimethylsiloxane moiety as the building block and known as Polydimethylsiloxanes (PDMS) have low viscosities, are stable at both end of temperatures, oxidative stable, and highly compressible. PDMS based compounds also have high dielectric stabilities. Their main constituting elements are silicon, oxygen, carbon, and hydrogen. A variety of functional PDMS compounds can be produced through hydrosilation reaction implying platinum and palladium catalysts [9]. PDMS group of compounds have the lowest glass transition temperatures, as the energy barrier required for the rotation of PDMS is virtually zero [10]. Looking at all these characteristics, different functionalized PDMS compounds were chosen for encapsulation in urea-formaldehyde shell wall to see the effect of functional groups on the encapsulation efficiency and properties of microcapsules.

## 2. Materials and Methods

### 2.1 Materials

Urea and formaldehyde (UF) were used as the shell wall forming monomers; gum Arabic (GA) and sodium dodecyl sulphate (SDS) were used as emulsifiers. Vinyl end polydimethylsiloxane (V-PDMS), hydroxyl end polydimethylsiloxane (OH-PDMS), and Epoxy end polydimethylsiloxane (E-PDMS) were used as core materials. Sodium hydroxide and citric acid were used for pH adjustment. Dried potassium bromide (KBr) was used for FTIR analysis. SDS was purchased from Sigma Aldrich. V-PDMS, OH-PDMS, and E-PDMS were purchased from Gelest Inc., USA. Remaining chemicals were purchased from Merck. All reagents were used without further purification. Figure 1 represents the chemical structures of functionalized PDMS taken from the catalogue “Reactive

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