

Synthesis and Characterization of Encapsulated 1-Tetradecanol for Thermal Energy Storage

K.Vijayrakesh, S.Muthuvel

Abstract: Microencapsulated phase change material (PCM) is used for industrial and house hold air conditioning system for longer term usage. In this study 1-Tetradecanol (C₁₄H₃₀O) is used as a core material. Urea (CH₄N₂O)-Formaldehyde (CH₂O) is used as shell material. Micro capsulation is made using in-situ polymerization technique. Prepared microcapsules were tested for feasibility to use cooling system. The average size was verified using Scanning Electron microscope (SEM) and result was found 3µm and shape was observed as spherical and surface morphology was smooth. The presence of chemicals of core and shell materials was verified by Fourier Transform Infra-Red spectroscopy (FT-IR). The peak temperature of microcapsules was found as 150°C from Thermo Gravimetric Analyzer (TGA). Thermal cyclic stability was verified by Differential Scanning Calorimetry (DSC). The cyclic temperature was observed as 45.9°C and 39.78°C. These prepared microcapsules can used to control the temperature of 37°C. This prepared microcapsule can be used for room air conditioning system

Keywords : Phase change material, Microencapsulation, 1-Tetradecanol, Urea-Formaldehyde shell, In-situ polymerization

I. INTRODUCTION

The present scenario shows that the energy consumption for everyday usage drastically increases due to increasing needs of air conditioning and refrigeration systems. To fulfil the need for energy more power sources are needed, such as atomic power plants, hydel power plants, thermal power plants and renewable power plants which involves huge investment and causes environmental pollution problems. The energy consumption varies across the day which makes the power plants unbalanced. High energy consumption is not only a problem for power plants but also it creates a large amount of emissions. Hence a deep research is required to balance the power requirement and reduce the environmental impact.

Energy utilization is mainly required for room air conditioning system, it is needed from an ordinary fan to till intelligent air-conditioning system. For example, in the food processing industries and pharmaceutical industries to preserve the quality of products a cooling environment required [2]. Hence, the refrigerators are used to fulfill these needs. Nevertheless, transporting the food products and medicines is highly complicated to maintain the same temperature [1]. Therefore the thermal environment of products should be maintained till it reaches the customers to maintain the quality. For transporting the products from one

to another place, various techniques are in use, such as dry ice cooling, vapor absorption, compression refrigeration, Passive cooling and peltier cooler [2].

To reduce energy utilization, passive cooling is suggested as one among the best options for maintaining the temperature. The passive cooling is also used as thermal latent heat storage for household and industrial usage [3]. In latent heat storage process, many methods are suggested, and the most commonly used methods are, pure phase changing material packs, the phase change material mixing metal particles for high thermal conductivity, form stable phase materials composites and encapsulated phase change materials [5][6][7]. Phase change materials have a disadvantage when heated more than melting temperature for a longer period it will unstable. That is, low heat transfer rate is available when used directly and after the its melts it cannot gain its shape again. If high thermally conductive metal powders is used, the sedimentation problem will be developed, which will reduce multicyclic usage with the same efficiency. Hence the encapsulation method is suggested for better use of PCM for a long period of time. But, to increasing thermal conductivity, the micro encapsulation method is suggested [7]. PCM capsules produced in various sizes such as macro encapsulation, micro encapsulation and nano encapsulation. In macro encapsulation, the surface area is less so heat transfer rate is also low due to less heat absorption, which reduces the efficiency of PCM. So micro encapsulation method is suggested [8].

Selecting a chemical polymerization method for microencapsulate preparation is a vital one, as it is highly chemically sensitive. Several methods were used for microencapsulation together with interfacial polymerization [11], complex coacervation [12] and emulsion polymerization [13][17], in-situ polymerization [14] and suspension polymerization [8][9]. Among several types of polymerization method, in this work, in-situ polymerization was adopted. In situ polymerization is widely used because this encapsulation method gives excellent diffusion tightness [16]. In in-situ polymerization method, microcapsules are formed by making shell material as emulsion and core material mixed in liquid form by heating unto its melting state. Then the emulsion will cover the core material due to the chemical reaction which forms the microcapsules and the size can be varied based on stirring speed and steering time.[12].

The study conducted by Fang et al.(2009)reported that Tetradecane [C₁₄H₃₀, Melting point 6°C and boiling point 257°C] as core material and urea formaldehyde as shell material also in-situ polymerization technique was used and reported that the formation of microcapsules [27].

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In the present study, 1-Tetradecanol (C₁₄H₃₀O) is used as core material which has a melting point of 37.5°C and boiling point of 260°C and urea formaldehyde (thermal conductivity 0.433 Nm-1K) was used as shell material because of high thermal conductivity next to water (0.599 Nm-1oC) [24]. This work focused upon synthesizing and characterization of urea-formaldehyde shell microcapsules containing 1-Tetradecanol as PCM for thermal energy storage to check possibilities of using on room air conditioning system. In this feasibility study, size was verified by SEM which is also used to verify the shape of microcapsule and surface morphology. To confirm the chemical present in the microcapsules, FT-IR characterization was carried out. DSC was used to study the heating and cooling cycle. To verify the thermal stability of microcapsule, TGA was used and results are discussed.

II. EXPERIMENTAL

A. Materials

1-Tetradecanol (C₁₄H₃₀O) was used as core material (purity - 95% from Alfa aesar, England), Urea (CH₄N₂O – 99%) and formaldehyde (CH₂O – 37-4% w/v) were used as shell material. Ethylene Malic Anhydride (C₂H₂(CO)₂O - EMA) was used as an emulsifier for the water-oil emulsion. Sodium hydroxide (NaOH) and concentrated Hydrochloric acid (HCl) is used for pH maintenance on the emulsion, Resorcinol (C₆H₄(OH)₂) is used as a cross-linking agent. Fang et al. (2009) reported various weight percentage of resorcinol and concluded that more than 10 percentages will not create any significant mass ratio on the microcapsules. Hence, the optimum weight percentage of 5 percentages is used in this study. Ammonium chloride (NH₄Cl) is used as a nucleating agent. Deionized water as laboratory reagent for dilution process was used in this study.

B. Selecting procedure

Various methods of encapsulation techniques have been already discussed. Hence in this work in-situ polymerization technique is selected, due to diffusion tightness [16]. The direct polymerization of a single monomer is carried out on the particle surface by an in-situ polymerization method.

C. Preparation of emulsion

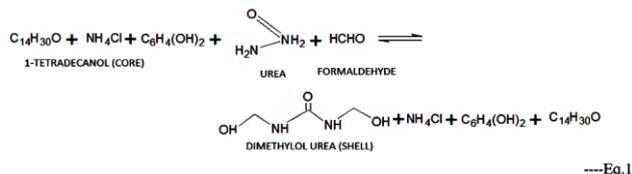
EMA solution was prepared for 2.5 weight percentages of 1-Tetradecanol (2.5 Weight % of PCM), 100ml distilled water and 2.35 gm. EMA were added and stirred at 500 rpm in magnetic stirrer until it changes to colorless condition and stored in an airtight test tube.

The following chemicals were added in 200 ml distilled water, materials and its proportions were 0.5 gm resorcinol, 0.5 gm ammonium chloride and 5 gm. urea. A 10 ml of prepared EMA solution was taken and added with the above prepared chemical mixture and the mixture was stirred at 500 rpm in a magnetic stirrer until the solution becomes colorless. The pH level was maintained between 3 and 5 by adding either sodium hydroxide or hydrochloric acid in the droplets based on the requirement the pH value adjustment.

D. Theory of microcapsules

In the prepared emulsion solution, 10 gm. of 1-Tetradecanol and 10 gm. of formaldehyde were added at 50°C, after the pH value adjustment. The stirring operation continued for four hours, maintaining the speed of 1000 rpm to give better surface morphology and smaller particle size [18][19][25]. The temperature of the solution was maintained

at 70°C. Hence, the material will become a solution, for the perfect encapsulation. Subsequent to stirring process, the solution was cooled to room temperature and the solution was filtered using vacuum membrane filter for filtering microcapsules. The process of microcapsule preparation and chemical reactions take place as shown in Eq1.



E. Theory of microcapsules

Microcapsules can be verified for various usage parameters, such as size, shape and surface morphology using SEM. For SEM test Scanning Electron Microscope EVO18 (CARL ZEISS), was used. Chemical presents can be verified using FT-IR test and suitable material spectrum can be checked using SHIMADZU-IR TRACER 100 and spectrum observed from 500 to 4000 cm-1 with resolution of 2 cm-1. Then the thermal stability can be verified using TGA 4000 and the instrument type is Pyris 6 TGA. The testing was done with sample mass about 7.568 mg with nitrogen flow of 20 ml / min and initial condition temperature about 30°C and the temperature rise programmed as 10°C/min. Temperature can be programmed for testing using the above-said instrument from 30°C to 500°C. The phase change properties of 1-Tetrodeconal were estimated by Differential Scanning Calorimetry test (PerkinElmer –Pyris DSC6000).

III. RESULTS AND DISCUSSION

A. Scanning Electron Microscope

It is necessary to find the shape and size of MEPCM, so it is very important to know the surface morphology and there should not be any dimples on the surface [18]. To find the shape, size and surface morphology, SEM was used. Since the surface morphology is considered as an important property for encapsulated phase change materials for better thermal stability on temperature change were also considered for strength. Wei li et al. (2011) investigated n-Octadecane with different copolymer shell materials for the surface morphology of different shell materials and different encapsulation methods and the experiment concluded that the thermal stability depends on surface morphology. Refat Al-Shannaq et al. (2015) investigated the effect of crosslink agent in microencapsulation strength and concluded that adding crossing linking agent will increase surface morphology and strength[20]. Guruprasad Alva et al.(2017) also reported that the nucleating agent can help to improve surface morphology by adding nucleating agents such as sodium chloride, ammonium chloride for better latent heat stability. In this work, ammonium chloride was used as nucleating agent and resorcinol used as crosslinking agent, Hence the result confirming smooth surface morphology on the microcapsule as shown in Figure. 1a. The Testing of free from buckles and dimples is an important consideration for smooth surface profiles[7], NihalSarier et al.(2007) reported using resorcinol as a cross-linking agent, encapsulation shape obtained as spherical and no dimples found on the surface for

the same urea-formaldehyde shell[21]. Huanzhi Zhang et al. (2009) also experimented with resorcinol as a cross-linking agent and ammonium chloride as a nucleating agent and reported that the surface morphology is good and shape obtained as sphere for melamine formaldehyde shell[15][22][23]. In this work, shape, size and surface morphology are shown in Figure 1(a), and (b). The experimental preparation shows that the size observed from 1 μm to 6 μm and shape of MEPCM is spherical and no dimples were found on the surface. The average size observed from particle distribution on the Figure 1. (c) of MEPCM is 3 μm . Hence, this result proved that the obtained shape is spherical, surface morphology is smooth without any dimples and average size is 3 μm .

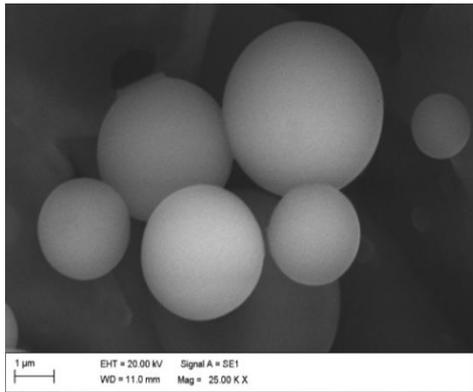


Fig 1(a)

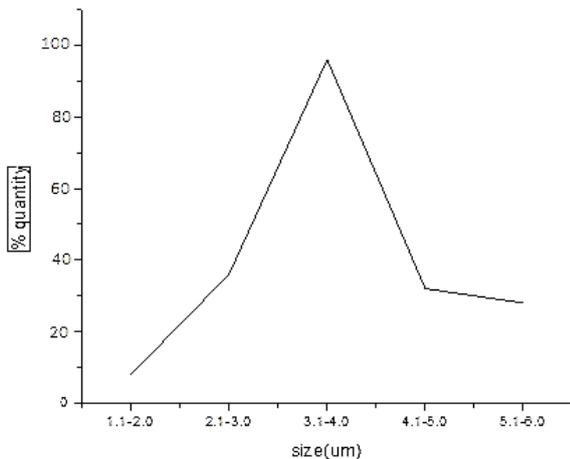


Fig 1(b)

B. Chemical characterization of the microcapsules

It is necessary to test the presence of shell and core material in the microcapsule. It is verified for the perfect formation of emulsification and encapsulation of phase change material. In this test all the material chemical group individual spectrum analyzed and the result can be verified by comparing the group spectrum on the final encapsulated phase changing material. Similar kind of testing was conducted and reported by various authors. [26], Song et al.(2015). Song et al.(2015) also microencapsulated 1-Tetradecanol with poly (methyl methacrylate) used as shell material and found the result for core material. Fang et al. (2009) used Urea-Formaldehyde as shell material and Tetradecane as a core material.

In this study, microcapsules were done by both 1-Tetradecanol and urea-formaldehyde spectrums. The absorption peaks values of 1-Tetradecanol, urea,

formaldehyde and the microcapsule are shown in Figure 2. From the FT-IR spectra lines of the microcapsules are available on both the peaks of 1-Tetradecanol and urea and formaldehyde. From Figure 2 it is also observed that the core and microcapsule absorption peaks at 1050 cm^{-1} to 1085 cm^{-1} is recognized in the stretching vibrations of C-O group and the absorption peaks at 718 cm^{-1} are recognized for stretching vibration in O-H group which is the characteristic absorption peaks of 1-tetradecanol.

The absorption peaks of Urea and Microcapsule at 1020 cm^{-1} to 1250 cm^{-1} is attributed to stretching vibration in C-N group from Figure 2, which is the absorption peaks found for Urea. Similarly, the absorption peaks of Formaldehyde measures from 1685 cm^{-1} to 1710 cm^{-1} and available in stretching in C=O lines which is the absorption peaks of Formaldehyde which is also shown clearly in Figure 2. This result shows the presence of 1-Tetradecanol, urea and formaldehyde in microcapsules. Hence the above results show the successful formation of microcapsules.

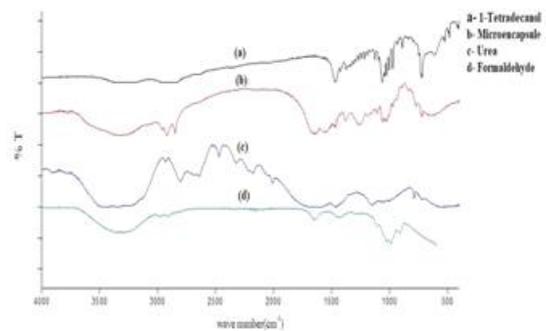


Fig 2. FT-IR (Core,Shell and microcapsule)

C. Thermogravimetric Analysis

The efficiency of MEPCM was tested for various thermal environmental conditions of degradation, because the thermally stable encapsulated phase change material can be used under safe temperature range. For this testing, Thermo Gravimetric Analyzer (TGA) is used. In this test, the programmed temperature rose to the sample and compared with the initial mass and changing mass due to evaporation of phase changing material at the temperature difference. Various researchers working on MEPCMs have tested for various temperatures, Song et al.(2015) tested TGA and found encapsulation degradation temperature of 100-175[4]. Fang et al.(2009) also tested with urea-formaldehyde as shell material and found supercooling temperature below 3°C. In this study, experimental procedure followed as per BS EN ISO 11358:1997 [11]. Figure 3 shows the TGA graph, It is observed that MEPCM starts to degrade from the temperature at 150°C and slowly reduced till it reached 170°C and significant weight loss started at 170°C and then it rapidly decreases its weight at 220°C and again it takes slow degradation at 250°C till it reaches 300°C. The weight loss shows less than 10 percentage. Figure 3 clearly shows that significant degradation temperatures are available between 150°C and 220°C. The result has been also shown in Table 1. Hence, The TGA result shows that the thermal stability of microcapsule of 1-tetradecanol as core material and urea-formaldehyde as shell material is good.

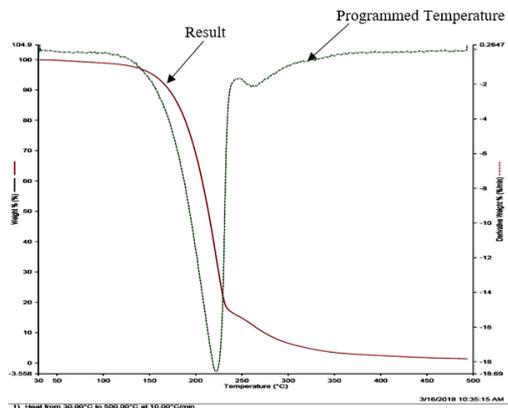


Fig 3 TGA graph

Table-I Comparison of Tetradecanol and Microcapsule

	Degradation Temperature	Mass loss
1-Tetradecanol	>100	100%
Microcapsules	100-150°C 150°C-220°C	3% 70%

B. Differential Scanning Calorimetry

The heating and cooling behavior of microcapsules is studied for better cyclic usage. For finding heating cooling or supercooling, Differential Scanning Calorimetry (DSC) is used for understanding the better usability of their MEPCM at various control temperatures. In this test, the given specimen heated and cooled in programmed temperature will increase and decrease.

In Figure 4 DSC curves show that at 45.9°C it shifted from 39.78°C for heating and cooling when encapsulation was made and the same peak and reduction obtained in heat flow in the MEPCM. It was also verified from JianguoZuo et al.(2011) for 1-Tetradecanol as pure phase changing material. Similarly, Song et al. (2015) reported for pure 1-Tetradecanol found to be heating and cooling latent heats are 210 J/g and 207 J/g but when poly(methyl methacrylate) microcapsules 1-Tetradecanol may 120.7 J/g and 118.4 J/g. Fang et al.(2009) also reported microcapsule using urea-formaldehyde as shell material for Tetradecane as the core material, supercooling temperatures was found at 3.29°C for pure tetradecane and 3°C for nanocapsules. So this experimental study shows that heating and cooling cycles of microcapsules with 1-tetradecanol as core material and urea-formaldehyde as shell material have better thermal stability at various controlled temperature.

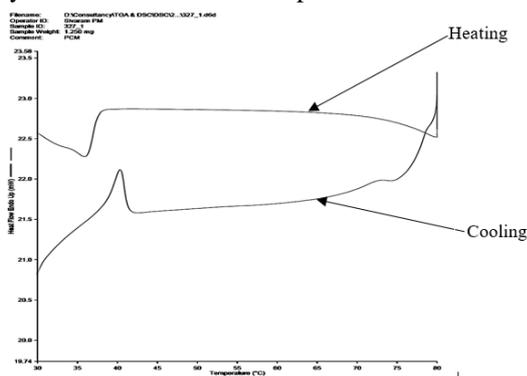


Fig. 4 DSC curve of MEPCM

III. CONCLUSION

1-Tetradecanol phase changing material successfully microencapsulated using urea-formaldehyde as shell material with proper control of speed at 1000rpm. The average size of microcapsules was found at 3µm and the surface morphology also conformed as smooth. The particle shape may be found as sphere. Materials for 1-Tetradecanol core and Urea-formaldehyde shell presents conformed using FT-IR by analyzing presents of individual spectrums of materials with spectrum of microcapsules. Thermal peak stability of microcapsules was tested and verified using TGA and the results found that the highest possible temperature of usage is about 150°C. Thermal cyclic change temperature stability verified was using DSC tests and found 45.9°C and 39.78°C for heating and cooling. Based on the results found from the study, the microcapsules formed successfully and it can be used for comfortable air conditioning system using heat thermal storage applications.

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