# Microencapsulation of Paraffin Wax Microspheres with Silver

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## ABSTRACT

Microencapsulation of phase change materials (PCMs) with metallic shell materials is a very innovative and challenging task. This can mitigate the problems related to thermal barrier for conventional nonconductive shell materials as well as enhance mechanical properties of PCM microcapsules. Such microcapsules can be integrated into microelectronic devices for their intermittent thermal management in mission critical components. The present work is aimed at developing a new method to synthesise phase change material encapsulated with metallic shell material and characterising the same. Paraffin wax microspheres were first synthesised and then encapsulated with silver through *in situ* chemical reduction. Further more, a new set of experiments were identified to analyse the quality of encapsulation. The thermal properties were investigated under differential scanning calorimeter and thermogravimetric analyser. The average diameter of paraffin wax microspheres (PW) is found to be ±329 μm. It reveals from DSC analysis that the enthalpy of fusion is minimum for PW@Ag-PVA amongst all others. Accordingly, higher deposition of Ag is possible for PW@Ag-PVA. This is also supported by TGA results where PW@Ag-PVA has only 40 per cent mass loss and the remaining samples have 100 per cent. However, even for PW@Ag-PVA the encapsulation is found incomplete. The present work provides knowhow of the difficulties associated with encapsulation of PCMs with metallic shell material.

**Keywords:** Microencapsulation; paraffin wax; Metallic shell material; Microelectronic thermal management

## 1. INTRODUCTION

Latent heat thermal energy storage (LHTES) system through phase change material (PCM) has become popular and attractive amongst the various energy storage techniques because it provides high energy storage density and stores thermal energy at a constant temperature, corresponding to the phase transition temperature of the energy storage materials. As PCM absorbs heat, it stores thermal energy by breaking internal chemical bonding, while it releases the stored energy by re-bonding when heat is discharged to surrounding[1]. Over the last few decades, effective use of PCMs has been proven in their utilisation in various thermal storage devices such as solar energy storage[2], space conditioning[3], industrial heat waste management[4], cooling technology for electronics[5,6], cold storage[7] and smart textiles[8] etc. Use of latent heat thermal energy storage devices is also found application in military services, where defence personnel are deputed in harsh environments[1].

Though several solid-liquid organic/inorganic chemicals are identified as PCMs, the majority of them fail to satisfy the adequate energy storage criteria for specific application either due to poor physical or thermal property. Amongst various solid-liquid PCMs, paraffin waxes (n-alkanes) are the most preferred by research community and commonly used for different applications because of their inherent properties such

as high latent heat storage capacity over a narrow temperature range, low volume change during phase transition, less super-cooling, good chemical and thermal stability, non-toxic and economically viable. Moreover, the latent heat capacity, as well as phase transition temperature of paraffin wax, is dependent on molar mass which can be controlled by suitable selection of the number of carbon atoms[9]. Therefore, there is flexibility in selecting paraffin wax as an appropriate PCM for specific applications by choosing the right number of carbon atoms. However, one of the major concerns associated with these solid-liquid PCMs is the leakage due to solid-liquid phase transition and its low thermal conductivity due to the fact that paraffin wax is polymeric in nature[9]. Appreciating the effectiveness of microencapsulation to mitigate the leakage problems associated with PCMs, in recent times several encapsulation methods and techniques have been developed by the researchers[10,11]. It is important to mention in this context that apart from solving leakage issue, microencapsulation has several features such as enhanced thermal conductivity of the encapsulated PCMs due to increased surface area to volume ratio, control dilation during phase transition, ease of integrability into any structural parts and protection from damaging environmental interaction[12]. However, the over-all thermal conductivity of microcapsules is still low due to polymeric shell wall materials. Microencapsulated PCM (MPCM) with ceramic[12,13] and polymeric[10] shell materials

have already been reported. It is important to mention in this context that the thermal properties of metals are higher than that of ceramics and polymers and accordingly metal coating encapsulation is one of the ways to improve the thermal characteristics of MPCM. Though inorganic shell materials are used to enhance thermal conductivity, employment of these characterises low encapsulation efficiency, longer cross-linking time and low mechanical strength[14]. Therefore, the notion of replacing the polymeric shell with metallic one is of utmost important and indeed involves a great novelty as well as a challenge. This replacement will not only improve thermal conductivity but also complement additional features such as good electrical conductivity and higher mechanical strength. Recently, a new method has been reported in the literature wherein poly (methyl methacrylate) (PMMA) PCM microcapsules were coated with silver using electroless coating technique. Using metal coating, the thermal conductivity of the PCM microcapsules is increased from 0.189 W/mK to 2.41 W/mK[15]. However, in this dual encapsulation, PMMA serves as high thermal resistance between PCM and silver shell coating. Therefore, there is a scope for further improvement in the thermal conductivity of microcapsule by encapsulating PCMs with metallic shell.

The aim of the present work is to synthesise phase change material encapsulated with metallic shell material and characterise the same. Unlike in polymeric encapsulation methods, the process of encapsulation is fragmented into two stages:

(i) Preparation of microspheres via emulsion technique, and
(ii) Encapsulation of microspheres with metallic shell via a chemical metallisation process.

Encapsulation of low temperature (<100°C) PCM by metals has not been reported so far. For the first time attempt has been made to encapsulate PCM with *Ag* to have high thermal conductive shell for enhanced heat flow. These encapsulated PCM is also expected to have high mechanical stability. Furthermore, it is important to point out that in most of the work reported so far, microencapsulation is done in single step process when PCM is in liquid phase. In single step processing, there is a possibility of PCM contamination by the shell materials depending upon the type of shell materials used[16]. Herein, encapsulation is done when PCM is in solid state and accordingly the chances of contamination is tried to be avoided to have MCPCMs with high heat storage efficiency.

## 2. MATERIAL AND METHODS

### 2.1 Material

Paraffin wax with melting temperature ($T_m$) of 58 °C - 60 °C, sodium hydroxide, dextrose extra pure and ammonia hydroxide solution 25 per cent were purchased from Merck Specialities Pvt. Ltd. (Mumbai). Silver nitrate (ACS grade), gum arabic (GA) and Sodium dodecyl sulfate (SDS) were purchased from Sisco Research Laboratories Pvt. Ltd. (Mumbai). Poly vinyl alcohol (PVA) was purchased from Loba Chemie Pvt. Ltd. (Mumbai). All reagents and solvents were of analytical grade and used as received from the suppliers.

### 2.2 Synthesis of Paraffin Wax Microspheres

An attempt was made to prepare microspheres through emulsification process using paraffin wax as oil phase, anionic SDS as a surfactant and in presence of distilled water. The block diagram depicting overall summary of the work is illustrated in Fig. 1. Initially, 250 ml of distilled water was heated to 80 °C and 6 wt.% of the SDS acting as a surfactant was added slowly. Molten paraffin wax (10 g) was added dropwise into it with stirring at 400 rpm in a magnetic stirrer. After the addition of molten paraffin wax, the prepared emulsion having micron size droplets of oil (coarse emulsion) was subjected to ultrasonication for 15 m (30 s on and 30 s off) at 45 per cent amplitude using a sonicator (Q700, Qsonica, USA) having a 12.7 mm titanium probe. Ultrasonication enables the paraffin wax to break down into smaller droplets. Solid paraffin wax microspheres were obtained by stirring the emulsion at 400 rpm to room temperature. The final product was obtained after filtration and washing with distilled water followed by drying at room temperature. Finally, size segregation of the microspheres was done using a sieve with mesh no. 20 having a nominal opening of 841 μm. The above mentioned process is schemed in Fig. 2.
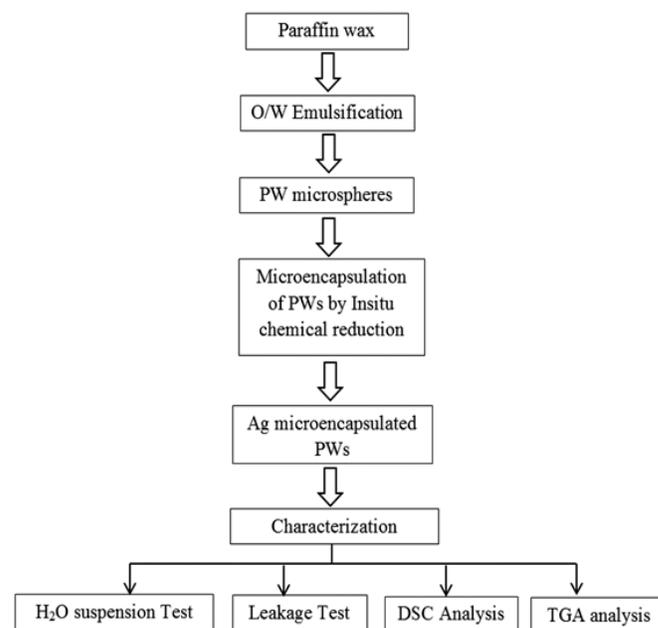


**Figure 1. Block diagram depicting overall summary of the work.**
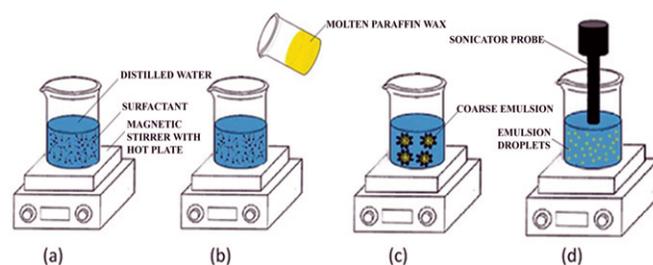


**Figure 2. Preparation of microspheres through emulsification (a) dispersion of surfactant, (b) addition of molten wax, (c) pre-emulsification, and (d) emulsification using sonicator.**

## 2.3 Encapsulation of Microspheres with Silver

Encapsulation of microspheres with silver is done through *in situ* chemical metallisation process. In this process, distilled water was first heated at 60 °C and 70 wt.% of gum arabic was added slowly as a dispersant under magnetic stirring at 400 rpm for 2 h. The solution was then cooled and centrifuged at 3000 rpm to remove insoluble particles, impurity from gum arabic. Thereafter, 0.30 g of paraffin microspheres was dispersed into the above prepared solution by stir mixing the suspension for 30 min at 400 rpm. Later, 0.5 g of Aqueous $AgNO_3$ solution and 0.6 ml of $NH_4OH$ (aq) were added and the resultant suspension was kept at 4 °C using a water chiller. 0.30 g of dextrose (reducing agent) and 0.17 g of NaOH added in the suspension as stirred at 400 rpm for 2 h. Finally, microcapsules were collected after filtration and DI water/ethanol washing using a centrifuge. Similar procedure was adopted to prepare encapsulated microspheres using PVA as a dispersant.

## 3. CHARACTERISATION

Optical micrographs of the paraffin wax microspheres (PW), PCM microcapsules with dispersant GA (PW@Ag-GA) and PVA (PW@Ag-PVA) were examined under Leica DM 2500M optical microscope. In order to verify the quality of encapsulation, three preliminary tests were performed. These are (i) water suspension test (ii) leakage test and (ii) Differential scanning calorimetry (DSC) analysis. The fundamental basis of water suspension test is that an object having a density lower than water will float when suspended in water. Here, paraffin wax microspheres will float when suspended in water due to its low density. On the other hand, microspheres encapsulated with metallic shell are expected to submerge. Further confirmation of proper encapsulation is harnessed from leakage test. Leakage test was done by keeping ~100 mg of the encapsulated microspheres in oven at 90 °C. The leakage was identified visually. Nevertheless, leakage test is not sufficient enough to confirm complete encapsulation of microspheres. This can be better predicted by DSC analysis. DSC analysis was performed using simultaneous thermal analyser (STA) 449 F3 Jupiter from Netzsch. 10 mg of sample was heated in alumina crucible from 30 °C to 180 °C at 10 °C/min in under $N_2$ purging. From the results, heating curve and latent heat storage capacity were determined and subsequently Enthalpy-Temperature was plotted. In order to investigate the thermal stability of the samples, TGA was done by taking ~10 mg of sample and heated from 30 °C to 500 °C at 10 °C/min with $N_2$ purging.

## 4. RESULTS AND DISCUSSION

### 4.1 Morphological Analysis

Emulsification is the process of blending two immiscible liquid with the application of shear force in presence of surfactant. The surface tension at oil-water interphase is reduced by absorbing surfactant on a dispersed phase droplets. Because of amphiphilic nature of SDS, the hydrophobic tail is absorbed by a paraffin wax droplet whereas negatively charge hydrophilic head surrounds the droplet. Accordingly, the electrostatic repulsion between the droplets accomplishes emulsion stabilisation by electrostatic repulsion[17]. The possible

mechanism is schemed in Fig. 3. Paraffin wax microspheres (PW) prepared by the emulsification process is as shown in Fig. 4(a). Optical examination reveals irregular bulging shaped PWs as the majority with few microspheres having a spherical morphology, as demonstrated in Fig. 4(b). The average size of the microspheres is determined as ± 329 μm. The deviation in the shape and size as hypothesised is possibly by Ostwald ripening effect during solidification. Small protruded surfaces on PWs as observed under optical microscope (Fig. 4(a)) again confirms Ostwald ripening effect nullifying the role of SDS. Furthermore, confluence between the tendency of paraffin wax to float on top layer and imbalance surface tension under stirring might have promoted the generation of the irregular bulging shaped microspheres. From Figs. 4(c) and 4(e), it can be found that encapsulated microspheres have dark brown and olive green colour respectively. This suggests successful deposition of *Ag* onto PWs also reported in other works[18]. However, morphology is found same as PWs as illustrated in Figs. 4(d) and 4(f) respectively. In addition, hair-like structure is seen in the case of PW@Ag-PVA. It could be due to attached PVA onto microcapsules even after washing with ethanol. Though these observations may be an indication of microencapsulation, one cannot be conclusive without further tests.
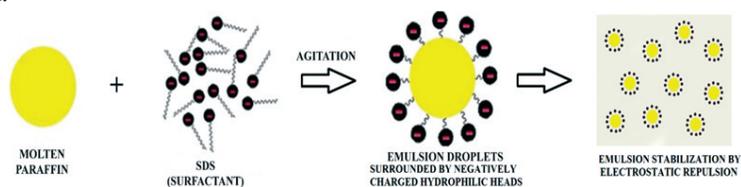


**Figure 3. Possible emulsification mechanism of paraffin droplets by electrostatic repulsion between negatively charged hydrophilic heads of SDS.**
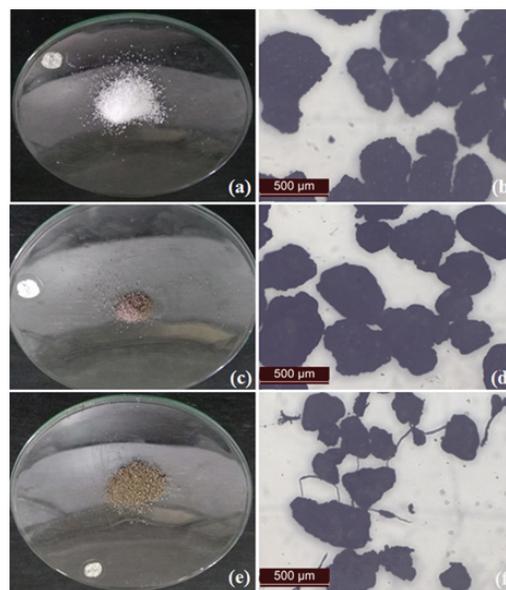


**Figure 4. Photograph of prepared (a) paraffin wax microspheres (PWs) (c) *Ag* encapsulated paraffin wax microspheres using PVA as dispersant (PW@Ag-PVA) (e) *Ag* encapsulated paraffin wax microspheres using GA as dispersant (PW@Ag-GA) and adjacent Figs. (b), (d), and (f) are their respective micrographs.**

## 4.2 Confirmation Tests for Encapsulation

In the present method *in situ* chemical reduction, paraffin microspheres were dispersed using a suitable dispersant and then metal salt is reacted with reducing agent to deposit metallic film on the surface of microspheres. The possible encapsulation mechanism of PWs with *Ag* using GA as a dispersant is presented in Fig. 5. It is important to mention in this context that GA not only serves as dispersant during encapsulation process but also provides a functional group to react with $Ag^+$ ions to form a complex compound. The $Ag^+$ nuclei on GA polymer chain serves as nucleation seeds for the growth of encapsulating film[19]. As mentioned earlier, three tests were conducted to verify the quality of encapsulation of the PCMs. Figure 6 demonstrates the water submersion test. It is observed that microcapsules are floating and hence, it can be concluded that *Ag* is not completely deposited on the surfaces of the PW microspheres. This observation is further confirmed by the leakage test as shown in Fig. 7. It was found that paraffin wax leaks out when heated beyond phase transition temperature.
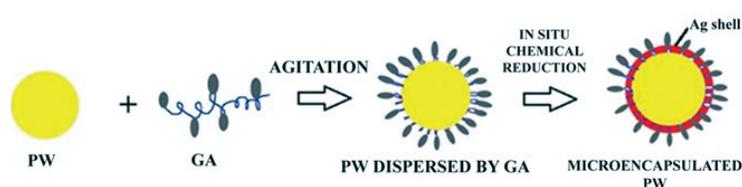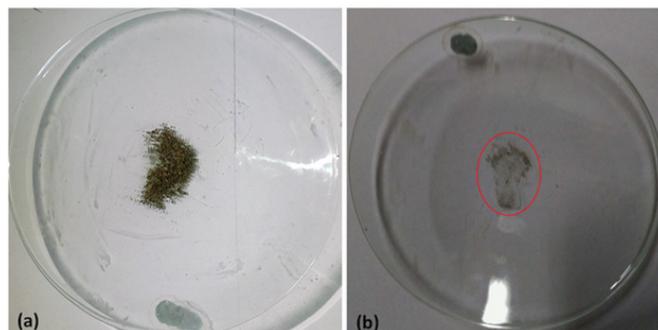


Figure 7. Leakage test (a) PW@Ag-PVA and (b) PW@Ag-GA.

Though it was established from the first two tests that *Ag* was incompletely coated, differential scanning calorimeter (DSC) was carried out as can be seen in Fig. 8. DSC scan was carried out only for melting and freezing cycle is not considered in the present work. This is considered for future study after successful encapsulation. Basically, DSC curve of PCM has characteristic two endothermic peaks. The first peak at low temperature is due to solid-solid transition of PCM whereas the second peak at phase transition temperature is due to solid-liquid transition[20]. The enthalpy of fusion is calculated by numerical integration of the area under the peak of thermal transition. Onset temperature was determined by drawing a line at the point of maximum slope of the leading edge of the DSC peak and extrapolating base line on the same side as the leading edge of the peak. MPCM will have lower latent heat of fusion than PCM since the former is having lesser amount of PCM. Figure 8(a) illustrates the DSC curve of pure paraffin wax with characteristic two endothermic peaks. The thermal properties of PWs and encapsulated paraffin are presented in Table 1. PWs have latent heat of fusion ~ 184.6J/ which is marginally lower than that of paraffin wax. The reason might be due to presence of unwashed surfactant used during the emulsification process. In the case of PW@Ag-GA, DSC curve do not show considerable changes in latent heat capacity as illustrated in Figs. 8(b) and 8(d), respectively. It has nearly the same enthalpy of fusion as that of sample PWs. Thus, confirming lesser amount of Ag deposition or Ag is not deposited on to PW@Ag-GA. On the other hand, for PW@Ag-PVA has an abrupt reduction in enthalpy of fusion ($\approx 37.5\%$) as compared to PWs as shown in Fig. 8(c) as well as Table 1. This behaviour might be due to the presence of larger amount of Ag onto PW@Ag-PVA. Figure 9 shows the variation of enthalpy with temperature for different samples. The rate of enthalpy of fusion is found increased between 40 °C to 80 °C for paraffin wax, PWs and PW@Ag-GA, accounted for limited restriction



Figure 5. Possible encapsulation mechanism of paraffin wax microsphere (PWs) by *in situ* chemical reduction of AgNO₃ using gum arabic (GA) as a dispersant.
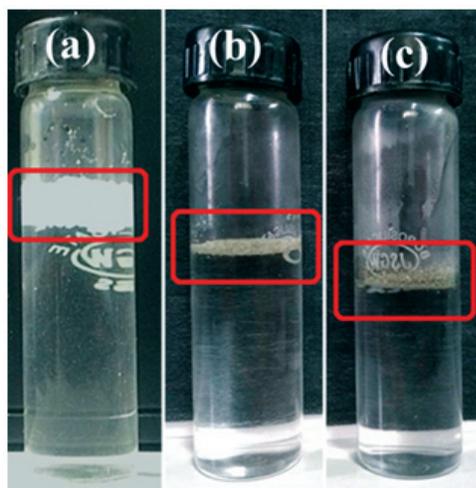


Figure 6. Water submersion test (a) PW (b) PW@Ag-PVA and (c) PW@Ag-GA.

**Table 1. Thermal properties of the samples**

| Samples | DSC | | | | TGA and DTG | | | |
|---|---|---|---|---|---|---|---|---|
| | $T_{onset}$ (°C) | $T_{peak}$ (°C) | $T_{endset}$ (°C) | $\Delta H_{fusion}$ (J/g) | $T_{onset}$ (°C) | $T_{max}$ (°C) | Residual mass (%) | Rate of decomposition (%/min) |
| Paraffin wax | 59.7 | 67.1 | 77.1 | 190.7 | 362.1 | 413.2 | 0 | 13.62 |
| PW | 60.9 | 70.1 | 81.3 | 184.6 | 363.1 | 412.7 | 0.76 | 12.98 |
| PW@Ag-PVA | 59.8 | 73.2 | 87.7 | 115 | 373.8 | 414.7 | 40.89 | 9.35 |
| PW@Ag-GA | 60.2 | 70.2 | 82 | 177 | 351.2 | 400.4 | 0.58 | 12.39 |
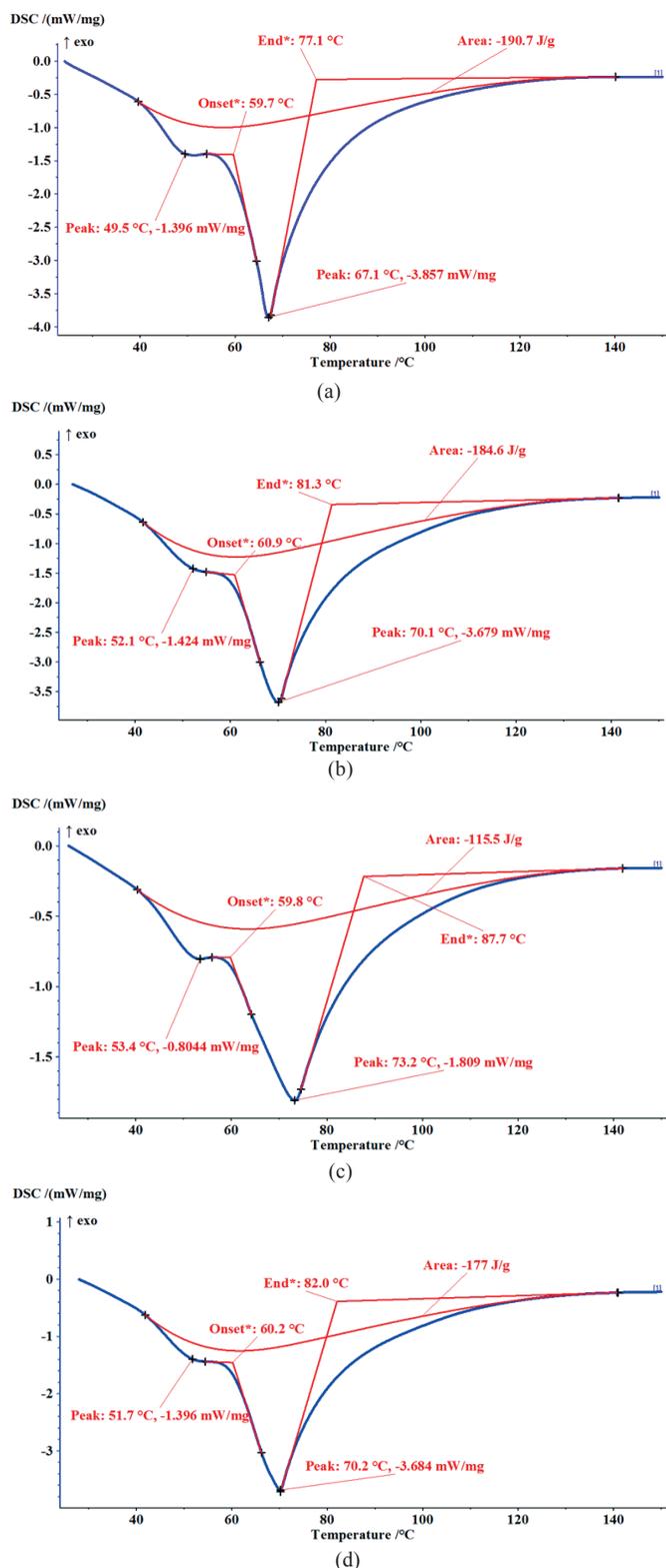
Figure 8.  DSC heating curves of the samples (a) Paraffin (b) PW (c) PW@Ag-PVA, (d) PW@Ag-GA.



Figure 9. The enthalpy-temperature curves of the samples.
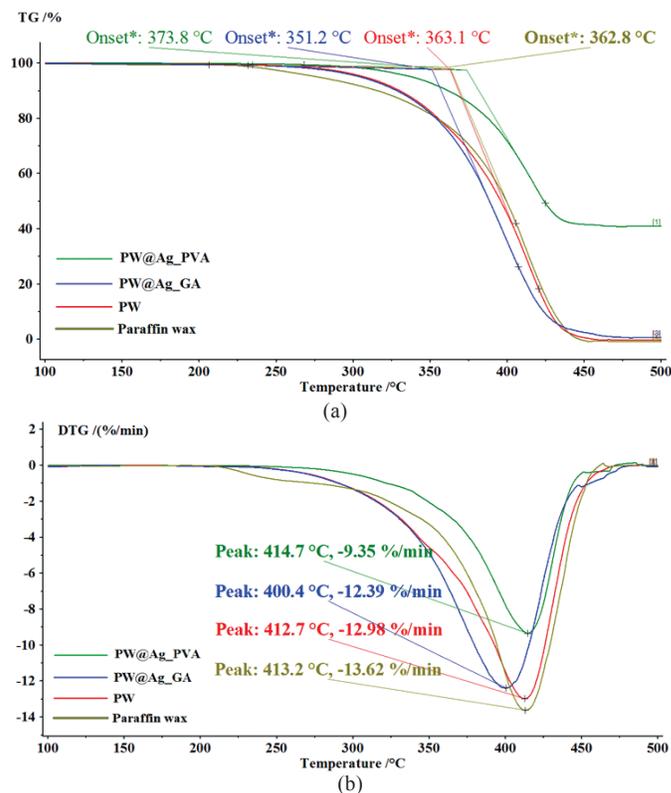


Figure 10. TGA (a) and DTG (b) curves of the samples.

to heat flow. This restriction is found enhanced for PW@Ag-PVA enabling reduced rate of enthalpy of fusion might be due to the presence of *Ag*.

Figure 10(a) depicts the thermal degradation behaviour of all the samples with increase in temperature. Other studies on TGA degradation beha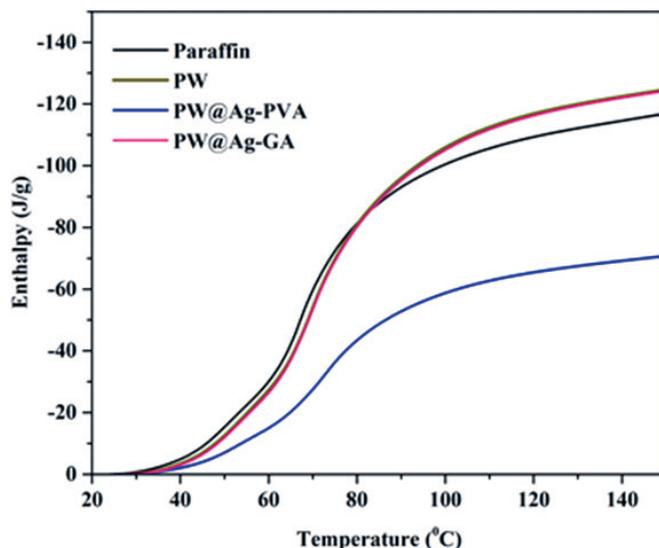viour of microencapsulated paraffin shows two-step decomposition primarily due to the evaporation of paraffin wax at lower temperature and then decomposition of shell material at higher temperatures[21]. However, in the present study microcapsules have only one distinguishable decomposition step. The probable reason is non-deposition or poor deposition of *Ag* on PWs. But, it is observed that sample PW@Ag-PVA has a mass loss of only 40 per cent. This suggests possibility of higher amount of *Ag* onto PW@Ag-PVA restricting the degradation of paraffin wax within the temperature range. Also, delayed degradation and lower rate of decomposition for PW@Ag-PVA is observed from DTG curve illustrated in Fig. 10 (b). Thus, it can be concluded

that *Ag* is deposited onto PW@Ag-PVA but incompetent to encapsulate completely. On the other hand, there was 100 per cent weight loss for other samples. This physically implies that deposition of *Ag* has not been achieved for PW@Ag-GA. One of the reasons may be the addition of $AgNO_3$ in aqueous form. It is interesting to mention here that $Ag^+$ ions may be repelled because of the hydrophobic nature of paraffin wax and it eventually restrains deposition on the paraffin microsphere. Another hypothesis is comparatively lack of surface activation[15] for PW microspheres. Therefore, problems in the encapsulation may be overcome by introducing $AgNO_3$ in non-aqueous form or alternatively providing sufficient surface activation using suitable bio-inspired catechol polymers[22].

## 5. CONCLUSIONS

A detailed experimental study was conducted to encapsulate phase change material with metallic shell material and characterised. Paraffin microspheres synthesised by emulsification technique were having irregular bulging shape and rough surface texture. This could be due to high density difference between the solvent and paraffin wax a well as Ostwald ripening effect during solidification. Further, these microspheres were encapsulated by *in situ* chemical reduction of metallic salt. Three tests were executed to examine the quality of encapsulation and thermal properties of the microencapsulated PCMs. The results indicated that microencapsulation is better for PW@Ag-PVA, but incomplete. Possible reasons may be due to repulsion between aqueous $Ag^+$ ions and hydrophobic paraffin wax microspheres or lack of surface activation of microspheres. The DSC and TGA thermographs indicated that the sample PW@Ag-PVA has higher deposition of *Ag* during encapsulation process whereas other samples do not. The outcomes of the present analysis may be helpful to successfully synthesise phase change material encapsulated with metallic shell material for the application in microelectronic thermal management system.

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In the current study, he is involved in the characterisation of the encapsulated PCMs.