

# Development of binary eutectic organic phase change materials for solar thermal energy storage systems



Anuradha Gupta, Kamal K. Kushwah, Sujeet K. Mahobia, V.V.S. Murty

**Abstract:** Solar thermal energy storage unit anchored fatty acids as Phase Change Materials (PCMs) having narrow range of transition temperature and high latent heat of fusion. In this paper, a new novel eutectic PCM was developed by using a fatty acid (acetamide) and non-paraffin organic PCM (acetanilide) for a sharp melting point and high latent heat of fusion. The optimized eutectic PCM may be used for middle temperature range solar thermal energy storage systems. The binary mixture of acetamide and acetanilide at various compositions by mass ratio (wt%) was prepared and optimized experimentally for lowest value of melting point at a eutectic mixture composition of 60 wt% of acetamide and 40 wt% of acetanilide. Eutectic PCM was analyzed by Differential Scanning Calorimetry (DSC) and Field-Emission Scanning Electron Microscopy (FE-SEM). DSC results revealed that optimized eutectic PCM has a sharp melting point of 65.37°C and high latent heat of fusion of 224.67 kJ/kg. Accelerated thermal cycle testing of optimized eutectic PCM was performed for 100 melting and freezing cycles and change in melting temperature and latent heat of fusion was acceptable.

**Keywords :** DSC, Eutectic, Latent heat thermal energy storage, Solar thermal energy.

## I. INTRODUCTION

Solar thermal energy storage became important due to the gap between demand and supply of solar energy. Out of the various possibilities, Latent Heat Thermal Energy Storage (LHTES) is attractive due to its unique property of accepting and releasing energy at a constant temperature called as melting temperature. In recent decades design and development of Thermal Energy Storage (TES) materials with appropriate thermal properties and their utilization in various renewable energy systems has drawn great attention across the world [1-3]. PCMs can be broadly grouped into two categories: organic compounds and salt-hydrates based products. Organic compounds have congruent melting, high latent heat of fusion, self nucleation, non-toxic,

recyclable nature, and no super cooling hence they are suitable for any application [4].

Organic PCMs which can be employed in low temperature applications are essentially fatty acids, which fulfill the most of the requirements. Among organic PCMs, fatty acid base PCMs possess better properties like low super cooling, high latent heat, non-toxic, good compatibility with container material, less volume change, better thermal conductivity, good thermal and chemical stability after repeated cycles [5-7], because of the protected carboxyl group [8]. Eutectic based on the fatty acids is of considerable interest due to its attractive properties [9]. Eutectic mixtures based on fatty acid were developed by Fauzi et al. [10] by using sodium stearate (SS), palmitic acid (PA) and myristic acid (MA). Optimized eutectic PCM MA/PA/SS and MA/PA PCMs were tested for 200, 500, 1000 and 1500 accelerated melting/freezing thermo-cycles. MA/PA/SS eutectic PCM has shown better thermal stability than MA/PA eutectic PCM over accelerated thermal cycle testing. In the literature Sharma et al. [11] accomplished accelerated thermal cycle test to understand the thermal stability of acetamide and concluded that acetamide was found to be thermally stable after 300 cycles. Buddhi et al. studied [12] the thermophysical properties of paraffin wax, stearic acid and acetamide over 1500 accelerated melting-freezing thermal cycles. The results showed that acetamide has good thermal stability and minor variation in melting temperature and latent heat over 1500 cycles. El-Sebaai et al. [13-14] performed 1000 thermal cycle testing of acetanilide and investigated its compatibility with container material. It was reported that acetanilide showed good thermal stability and good compatibility with aluminum container. The selection criterion for any phase change material is mainly based on the latent heat of fusion and melting/freezing point. For a particular application, a tailor made PCM having suitable melting temperature is necessitated. In our study acetamide and acetanilide were taken as the component materials for the development of the novel eutectic PCM composition because they have sharp melting point, high thermal and chemical stability, high latent heat of fusion, low cost, non-toxic and available in large. The optimized PCM was studied for thermal stability over accelerated melting and freezing cycles. Thermo-physical properties and surface morphology of optimized eutectic PCM were studied by DSC and FE-SEM. The thermo physical properties of independent acetamide and acetanilide are shown in the Table I.

**Revised Manuscript Received on December 30, 2019.**

\* Correspondence Author

**Anuradha Gupta\***, Physics Department, Govt. Auto. Holkar Science College, Indore, India.

**Kamal K. Kushwah**, Applied Physics Department, Jabalpur Engineering College, Jabalpur, India.

**Sujeet K. Mahobia**, Applied Physics Department, Jabalpur Engineering College, Jabalpur, India.

**V.V.S. Murty**, Physics Department, Govt. Auto. Holkar Science College, Indore, India.

© The Authors. Published by Blue Eyes Intelligence Engineering and Sciences Publication (BEIESP). This is an [open access](https://creativecommons.org/licenses/by-nc-nd/4.0/) article under the CC-BY-NC-ND license <http://creativecommons.org/licenses/by-nc-nd/4.0/>

## II. EXPERIMENTAL METHOD

### A. Preparation of binary eutectic mixture

**Table- I: Thermo-physical properties of component PCM**

Property	Acetamide	Acetanilide
Melting Temperature, °C	82	118.9
Latent Heat of Fusion, kJ/kg	263	222
Specific heat, kJ/kg°C	1.94	2

**Table- II: Eutectic binary mixture of PCMS at various mass ratios of acetamide and acetanilide.**

Sample no.	Mass ratio (wt%)	
	Acetamide	Acetanilide
1	45	55
2	50	50
3	55	45
4	60	40
5	65	35
6	70	30

In our experimentation, eutectic mixture of acetamide (Merck, 99.0%) and acetanilide (Merck, 99.0%) was prepared in various compositions by wt%, under normal atmospheric conditions by melting-mixing method. The melting point and freezing points at every composition is noted for the analysis. The composition of acetamide was varied from 45 wt% to 70 wt% in the binary eutectic mixture of acetanilide and acetamide with different compositions as shown in Table II.

### B. Accelerated thermal cycle test

The applicability of any PCM in a specific solar TES system depends on performance of PCM during charging and discharging cycles. The thermal energy release and absorb by a TES system, must be at same temperature during each cycle. For long and continuous use of system consistency in melting temperature and latent heat is required over maximum possible number of accelerated thermal cycles. Hence accelerated thermal cycle testing is essential requirement before applying any PCM in a suitable specific application because durability of TES system depends upon the thermal stability of PCM over accelerated thermo-cycles.

To investigate the thermal stability of optimized eutectic composition of acetamide and acetanilide, accelerated thermal cycle test was performed. First eutectic PCM was heated till melting completely and then cooled down till it solidifies completely and the optimized eutectic PCM has made to gone through 100 such heating/cooling cycles.

## III. CHARACTERIZATION TECHNIQUES

### A. DSC analysis

The DSC [Mettler Toledo] analysis was used to measure the thermal characteristics of the eutectic mixture containing varied composition of acetamide and acetanilide in order to obtain optimized composition. For DSC analysis samples (3.9-6 mg) were weighed in a sealed aluminium pan and an empty pan was used for reference. DSC analysis was performed from 25-100° C with a heating rate of 10° C per minute. The melting point of the various eutectic mixtures was calculated. Cycled and non-cycled optimized eutectic PCM was analyzed by DSC and thermophysical properties before and after cycle testing were reported and analyzed.

### B. FE-SEM analysis

The surface morphology of optimized acetamide/acetanilide eutectic PCM was analyzed by using FE-SEM [Supra 55 Zeiss] characterization technique.

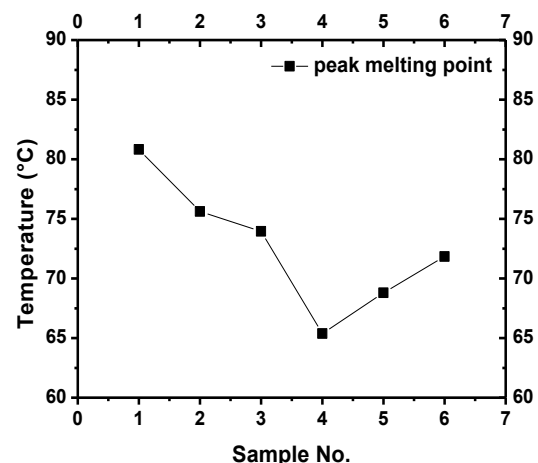
## IV. RESULT AND DISCUSSION

### A. Investigation of eutectic point

Eutectics having various wt% of acetamide and acetanilide were prepared and their melting and freezing cycles are analyzed. A known composition of acetamide and acetanilide by wt% was considered and its thermal performance was studied. It was observed that the peak melting temperature of the binary eutectic mixture varies with the wt% of the independent component. Variation of peak melting temperature for various prepared eutectic mixture samples are as shown in Fig. 1. As the amount of the acetanilide decreases and the amount of acetamide increases in the eutectic mixture, the melting temperature range decreases and a sharp lowest melting point 65.37 °C was observed for sample 4 at a composition of 60 wt% of acetamide and 40 wt% of acetanilide, after this melting point increases. It is depicted from the results that 60/40 is optimized mass ratio for acetamide/acetanilide eutectic PCM. It is observed that the melting point of the eutectic mixture is less than the melting point of the independent components of the eutectic mixture.

### B. DSC analysis

Out of the different compositions of acetamide and acetanilide, eutectic mixture having composition of 60 wt% of acetamide and 40 wt% of acetanilide has a melting point of 65.37° C was optimized and its DSC curve is shown in Fig. 2. The optimized eutectic temperature was found to be suitable for middle temperature range solar thermal storage applications. The latent heat of fusion of optimized eutectic was found to be 224.67 kJ/kg.



**Fig. 1. Variation of peak melting temperature of acetamide/acetanilide mixture.**

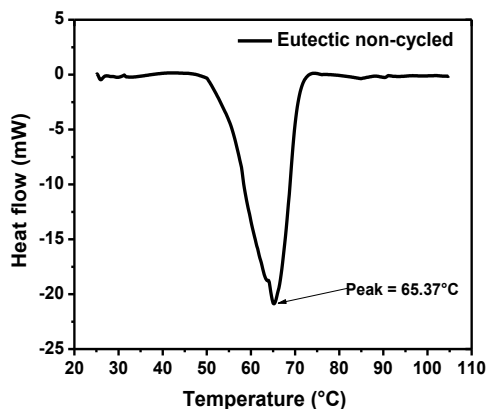


Fig. 2. DSC curve of optimized eutectic of 60 wt% of acetamide and 40 wt% of acetanilide before cycle testing.

### C. Thermal stability of optimized eutectic

The DSC curve of optimized acetamide/acetanilide eutectic before and after 100 accelerated thermo-cycles are as shown in Fig. 2 and Fig. 3 respectively. DSC analysis results revealed that after 100 accelerated thermo-cycles peak melting point and latent heat of melting of optimized eutectic PCM was found to be 64.95 °C and 223.29 kJ/kg respectively. The latent heat of fusion after 100 thermo-cycles altered by -1.38 kJ/kg and the peak melting temperature altered by -0.42 °C. The variation in latent heat of melting and melting temperature is acceptable. The results revealed that over 100 accelerated melting/freezing cycles the developed acetamide/acetanilide eutectic has shown good thermal stability.

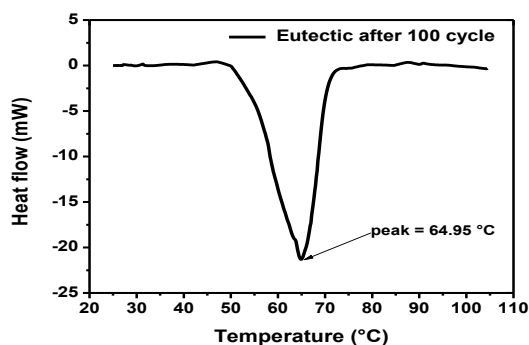


Fig. 3. DSC curve of optimized eutectic of 60 wt% of acetamide and 40 wt% of acetanilide after cycle testing.

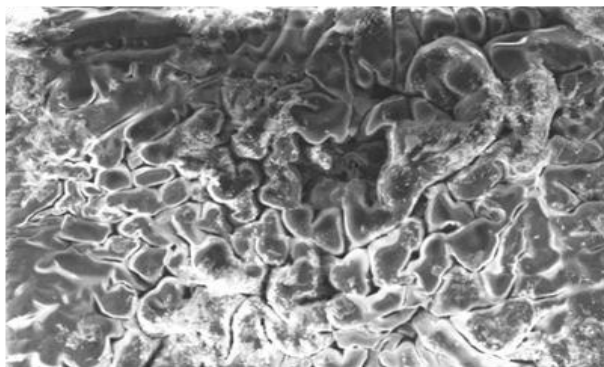


Fig. 4. Fe-SEM micrographs of optimized eutectic PCM with 60 wt% of acetamide and 40 wt% of acetanilide.

### D. Surface morphology of optimized eutectic

The FE-SEM image of optimized acetamide/acetanilide eutectic PCM is as shown in Fig. 4. It was observed that eutectic was showing flake like layer structure with a milky colour. Fe-SEM micrograph confirms the uniform distribution and complete mixing of acetamide and acetanilide in eutectic PCM.

### V. CONCLUSION

In this paper, a new eutectic mixture containing novel organic PCMs acetamide and acetanilide at various compositions by wt% was developed. Samples were analysed by DSC in order to determine optimized composition of component PCMs. Results revealed that a sharp melting point of 65.37 °C with a high latent heat of melting of 224.67 kJ/kg was observed at optimized eutectic mixture composition of 60 wt% of acetamide and 40 wt% of acetanilide. Surface morphology of optimized acetamide/acetanilide eutectic was analyzed by using FE-SEM and it confirmed homogeneous mixing of acetamide and acetanilide. After 100 accelerated melting/freezing cycles optimized eutectic PCM has shown good thermal stability with acceptable change in melting point and latent heat of melting. Hence acetamide/acetanilide eutectic has shown a great potential for being used in any moderate range solar thermal energy storage application.

### ACKNOWLEDGMENT

The authors are thankful to Dr. Suresh T. Silawat, Dr. R.C. Dixit and Dr. G. D. Gupta Govt. Autonomous Holkar Science College, Indore, India for their encouragement and providing facilities for experimentation. The authors are also grateful to Dr. M. Awasthi and Dr. Suresh Bhardwaj, UGC DAE CSR, Indore and Kinney Pandey, IIT Indore for their support in characterization of the samples and useful discussions.

### REFERENCES

1. R. Jacob, M. Belusko, M. Liu, W. Saman and F. Bruno, "Using renewables coupled with thermal energy storage to reduce natural gas consumption in higher temperature commercial/industrial applications", *Renew. energy*, vol. 131, 2019, pp.1035-1046.
2. G. Li, and X. Zheng, "Thermal energy storage system integration forms for a sustainable future", *Renew. Sustain. Energy Rev.*, vol. 62, 2016, pp.736-757.
3. J.P. Da Cunha and P. Eames, "Thermal energy storage for low and medium temperature applications using phase change materials—a review", *Appl. Energy*, vol. 177, 2016, pp.227-238.
4. P. Kauranen, K. Peippo and P.D. Lund, "An organic PCM storage system with adjustable melting temperature", *Solar Energy*, vol. 46, no. 5, 1991, pp.275-278.
5. G.J. Suppes, M.J. Goff and S. Lopes, "Latent heat characteristics of fatty acid derivatives pursuant phase change material applications", *Chem. Eng. Sci.*, vol. 58, no. 9, 2003, pp.1751-1763.
6. S. Kahwaji, M.B. Johnson, A.C. Kheirabadi, D. Groulx and M.A. White, "Fatty acids and related phase change materials for reliable thermal energy storage at moderate temperatures", *Sol. Energy Mater. Sol. Cells*, vol. 167, 2017, pp.109-120.
7. A. Sari and K. Kaygusuz, "Some fatty acids used for latent heat storage: thermal stability and corrosion of metals with respect to thermal cycling", *Renew. Energy*, vol. 28, no. 6, 2003, pp. 939-948.
8. D. Rozanna, T.G. Chuah, A. Salmiah, T.S. Choong and M. Sa'ari, "Fatty acids as phase change materials (PCMs) for thermal energy storage: a review", *Int. J. Green Energy*, vol. 1, no. 4, 2005, pp.495-513.



9. D. Feldman, M.M. Shapiro, D. Banu and C.J. Fuks, 1989. Fatty acids and their mixtures as phase-change materials for thermal energy storage”, Sol. energy mater., vol. 18, no. 3-4, 1989, pp.201-216.
10. H.Fauzi, H.S. Metselaar, T.M.I. Mahlia and M. Silakhori, “Thermo-physical stability of fatty acid eutectic mixtures subjected to accelerated aging for thermal energy storage (TES) application”, Appl. Thermal Eng., vol. 66, no. 1-2, 2014, pp. 328-334.
11. S.D. Sharma, D. Buddhi and R.L. Sawhney, “Accelerated thermal cycle test of latent heat-storage materials”, Solar Energy, vol. 66, no. 6, 1999, pp.483-490.
12. Atul Sharma, S.D. Sharma and D. Buddhi, “Accelerated thermal cycle test of acetamide, stearic acid and paraffin wax for solar thermal latent heat storage applications”, Energy Convers. Manag., vol. 43, no. 14, 2002, p.p. 1923-1930.
13. A.A. El-Sebaei and F. Al-Agel, “Fast thermal cycling of acetanilide as a storage material for solar energy applications”, J. Sol. Energy Eng., vol. 135, no. 2, 2013, p.p. 024502.
14. A.A. El-Sebaei, S. Al-Amir, F.M. Al-Marzouki, A.S. Faidah, A. Al-Ghamdi and S. Al-Heniti, “Fast thermal cycling of acetanilide and magnesium chloride hexahydrate for indoor solar cooking”, Energy convers., Manag., vol. 50, no. 12, 2009, pp.3104-3111.

### AUTHORS PROFILE



**Mrs. Anuradha Gupta** is pursuing her Ph. D. in Govt. Auto. Holkar Science College, Indore (M.P.). Her area of research is dye sensitized solar cells and material development for solar thermal energy storage systems. She has published good number of papers in national and international conferences and three papers in various reputed journals.



**Dr. Kamal Kumar Kushwah** Presently working as an Assistant Professor in Department of Applied Physics, Jabalpur Engineering College, Jabalpur, Madhya Pradesh, India. He has published 26 research papers in National and International journals. He was conferred upon with “Best Faculty” and “Rajya Gaurav Samman 2018”. He has authored two Book chapters on Luminescence and Photovoltaic technology. He holds a high place in the field of Nanoscience and Nanotechnology internationally and nationally. He is also involved with the popularization of science through Indian Association of Physics Teachers (IAPT) and Anveshika Vigyan Prasar Yojna running by IIT Kanpur.



**Dr. Sujeet Kumar Mahobia** Dr. Sujeet Kumar Mahobia has completed his Ph.D. from Rani Durgavati Vishwavidyalya, Jabalpur, Madhya Pradesh (MP) India. Presently he is working as Asst. Professor in Department of Applied Physics, Jabalpur Engineering College, Jabalpur, M. P. He is doing research in material science and has published 15 research papers in reputed National/ international journals. He has 13 years teaching experience.



**Dr. V.V.S. Murty** is currently working as a Professor of Physics in Government College of Madhya Pradesh, India. He has guided six Ph. D. students and presently working on Plasmonic dye sensitized solar cells and material development for solar thermal energy storage systems. He has published good number of papers in various reputed journals and conferences.