# EXPERIMENTAL INVESTIGATION AND NUMERICAL SIMULATION OF CARBOXYLIC (COOH) GROUP FUNCTIONALIZED GRAPHENE-BASED PARAFFIN WAX AS PHASE CHANGE MATERIAL IN A THERMAL ENERGY STORAGE SYSTEM

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## SUMMARY

This study analyses the melting time and liquid fraction during melting of carboxylic (COOH) group functionalized Graphene-based paraffin wax (PW) Phase Change Material (PCM) inside a thermal energy storage system (TESS) both experimentally and numerically (using Ansys Workbench 15.0 academic version). Its performance in terms of time taken for melting during charging/melting analysis is compared numerically and experimentally which leads to a good conformance and validates this research. The average error in the melting times of experimental and numerical result is found to be 1.9 %, 2.0 % and 2.6 % for three different flowrates which shows a very good agreement between experimental and numerical simulation results.

## KEYWORDS

Numerical simulation, Experimental heat transfer, Thermal energy storage system, COOH functionalized graphene

## 1. INTRODUCTION

Globally, in developed and developing countries as the economy is progressing to higher levels, the demand for energy supply is also showing an upward trend and increasing exponentially. The population explosion in developing countries has led to the search of new energy resources as the reliance on non-renewable source of energy and fuels (fossil) needs to be lessened. Also due to rising pollution levels the scientists are finding new ways to depend upon sources of cleaner energy production. The focus of this time is on PCMs which can store the surplus amounts of energy and the stored energy can be utilized in the hours of need. Such energy comes under the umbrella of latent heat thermal energy storage systems which stores tremendous amounts of energy during phase change. It is different from sensible heat storage systems that stores energy by changes in internal energy and not by change of phase. Scientists have proven that latent heat storage is better than sensible heat storage by storing energy 4 to12 times more energy (Nagar et al., 2022).

A great deal of experimental work and its corresponding numerical simulations are needed to find materials that can become energy storage materials in this era. The experimental work can be carried out on TESS having PCMs as organic PW and fatty acids etc. doped with higher conductivity materials like functionalized graphene and functionalized carbon nano tubes (CNT) etc. The functionalized graphene and CNT are stated because functionalization increments the properties of pristine materials in all ways like increment in chemical, thermal and mechanical properties. Even the dispersibility in organic PCM increases due to addition of functional group to the pure materials. Although pure PCM suffers from lower thermal conductivity the addition of functionalized graphene (Kumar et al., 2021) improves this property. In this work the author has used PW as PCM which is doped by COOH group functionalized graphene for the above stated reasons.

Firstly, the PW is procured along with COOH group functionalized graphene and the experimentation is done

inside a TESS after doping COOH functionalized graphene in PW inside the PCM cylinder of TESS. The time taken to melt/charge is recorded for different percentages of COOH group functionalized graphene after doping it in PW in amounts of 0.25 Vol %, 0.50 Vol %, 0.75 Vol %, 1 Vol % at three different flow rates of HTF (6.25 ml/sec, 12.5 ml/sec and 25 ml/sec) which passes through the copper heat exchanger and transfers heat to the PCM doped with nano particles surrounding the helical coiled heat exchanger inside the PCM cylinder. The results so obtained are validated and checked by ANSYS Fluent 15.0 by making the model, inputting the boundary conditions and solving it with the help of a Ansys 15.0 solver. The Ansys 15.0 post processor helps in visualizing the results. The deviation in experimental time and numerical simulations time is noted and supported with valid reasons (Nagar et al., 2022).

Some relevant literature review done by varied researchers would also be discussed in this section. Pandey et al. (Pandey et al., 2021) used building energy simulation tools (EnergyPlus) and Computational fluid dynamics tools (Ansys Fluent) for understanding the effects of energy demand and thermal comfort on built in environment and in initial design stages for hard conditions of climate. They used co-simulation methodology by using both building energy simulation tools (EnergyPlus) and Computational fluid dynamics tools (Ansys Fluent). Singh et al. (Singh et al., 2021) used eutectic salt PCM doped with COOH group functionalized grapheme (Kumar et al., 2020) in the weight fraction ratio of 1% to 5% and determined the micro structure using scanning electron microscopy (SEM). The thermo physical properties were also determined using differential scanning calorimetry (DSC) and thermal conductivity tests by laser flash analysis (LFA) method. Talebizadehsardari et al. (Talebizadehsardari et al., 2021) used plate type heat exchanger having zigzag configuration in their work. Simulation model was prepared and verified with other literatures for charging and discharging of the PCM. The various parameters like average temperature of heat transfer fluid (HTF), zigzag angle and inlet flow rate proved that the storage performance increased considerably due to such factors working in unison. Karthikeyan et al. (Karthikeyan et al., 2021) performed Computational fluid dynamics study on two encapsulated PCM, namely Paraffin wax and D-Mannitol by using ANSYS 2019R2. Transient numerical analysis was done on isothermal wall considering both Boussinesq approximation and gravity effects in the numerical procedure. Shaker et al. (Shaker et al., 2021) put his efforts towards solar applications of encapsulated PCMs due their high energy storage capability. The numerical work was validated by corresponding experimental model taken from the various literatures. The different parameters considered were solidification temperature, number of fins, HTF temperatures and HTF flow rate etc. The two PCMs compared in their work were Lauric acid and PW. By increasing the flow rate and number of fins they directly got the result as decrease in melting time for both the considered PCM.

Although substantial research have been done on LHTES system using paraffin wax (PW) PCM but the experiments done on TESS (Nagar et al., 2022) used in this research has never been simulated by Ansys Fluent workbench software thereby proving the novelty of this research. The concentric cylinder used in this research has a centrally placed Cu made helical heat exchanger inside which the HTF (water) flows. Surrounding the PCM (PW) and enclosing the Cu made helical heat exchanger is a stainless steel (SS) pipe. The interface between the helical heat exchanger and SS pipe is filled with COOH group functionalized graphene mixed with PW. The warm HTF heated from the source tank by electric heaters flows inside the Cu made heat exchanger transferring the heat to the solidified nano particle based PCM. By this the solidified PCM absorbs energy and melts thereby storing large amount of energy which can be released during time of need. To minimize heat losses to the environment foam based thermal insulation is provided over the SS pipe. The whole structure is placed inside the Galvanized Iron cylindrical tank. For efficient charging as the concentration of COOH group-based graphene in the PCM increases the melting time decreases. This has been proved and validated by the simulations done on Ansys Fluent 15.0 Workbench software.

## 2. OBJECTIVE OF THIS STUDY

The main objective of this experimental and numerical study was to find out the amount of time taken in minutes to completely charge/melt the advanced PCM (functionalized graphene (Kumar et al., 2019) mixed with PW) which absorbs energy for melting in the PCM cylinder of TESS. As the concentration of COOH group based functionalized graphene increases from 0.25% to 1% in steps 0.25 volume % in basic PCM which is PW, the charging time decreases as provided by the solidification/melting module of ANSYS Fluent 15 academic version. The charging time also decreases with increment in flow rate (6.25 ml/sec, 12.5 ml/sec and 25 ml/sec) of the HTF due to rapid heat transfer phenomenon. Also, the other aim is to, visualize numerically the liquid fraction of the advanced PCM with respect to time. The experimental charging times were validated with times of melting done by numerical simulation performed on ANSYS workbench 15.

## 3. MATERIALS USED, PURCHASE AND PREPARATION

The usage of PW (PCM) as a thermal energy storage material can be understood by taking a look in to its material properties. They suggests the higher latent heat of melting (153.05 kJ/kg) and higher heat storage properties. To counter the effect of poor thermal conductivity of pristine PW, it is doped with COOH group functionalized

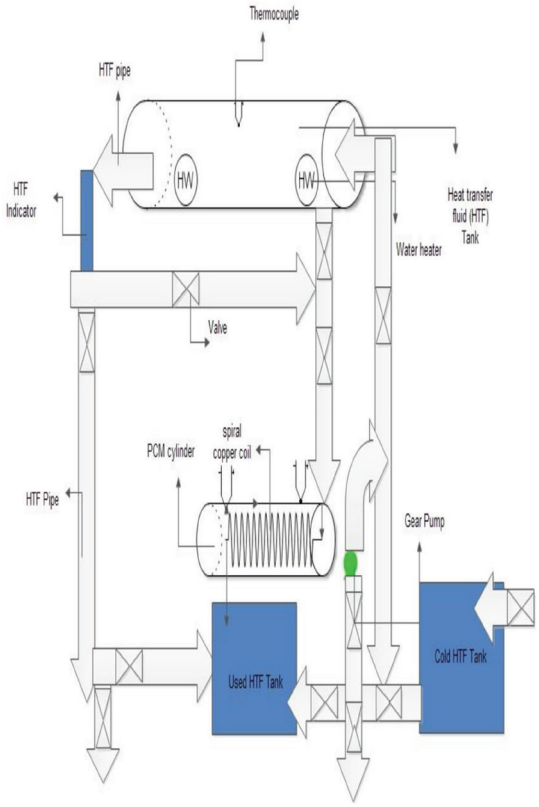



Figure 1. Schematic diagram of Thermal energy storage system (Nagar et al., 2022)

graphene (Nagar et al., 2022). The purchase of material was done according to (Nagar et al., 2022).

The hybrid PCM (PW+ COOH group functionalized graphene) was prepared first by weighing and melting PW and then adding COOH group functionalized graphene in volume percentages of 0.25, 0.5, 0.75 and 1% to the melted PW and stirring using magnetic stirrer.

## 4. BASIC EXPERIMENTATION

The basic schematic diagram and real photograph of thermal energy storage systems (TESS) is as shown in figure 1 and figure 2, respectively. The authors are using Just one PCM cylinder in this experimentation. The experimentation is similar to (Nagar et al., 2022).

In general, a trend obtained was noticed which signifies that higher the % of COOH group functionalized graphene in PW lesser would be the charging times. Also, as the flow rate of HTF was increased the graphene took lesser time to melt. This was due to the fact that COOH group functionalized graphene had very high thermal conductivity because of the free pi–electron on the carbon atoms in its structure and very high electron mobility. The

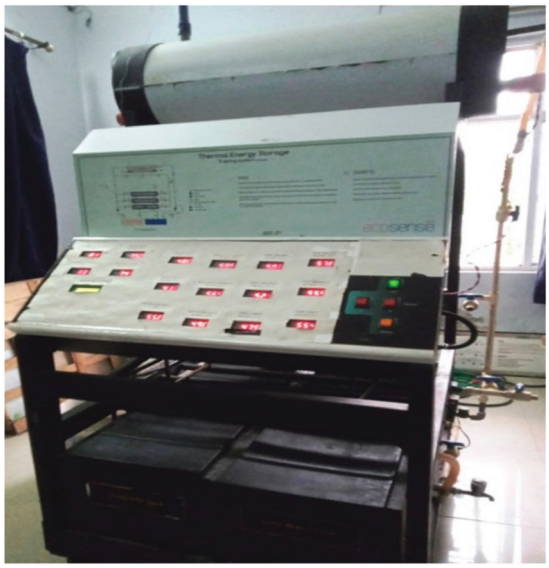


Figure 2. Photograph of thermal energy storage system (Nagar et al., 2022)

fourth carbon atom on the graphene structure becomes delocalized and acts mainly as a carrier of charges.

## 5. NUMERICAL METHODOLOGY

### 5.1 SIMULATION AREA, GEOMETRY, DISTRIBUTION OF GRIDS AND ITS INDEPENDENCE AND MESHING

As per figure 3 (a), the helical heat exchanger shown in figure 3 (b) was placed inside the Stainless-steel (SS) cylinder. The warm water which is the HTF (Heat Transfer Fluid) at inlet was coming from the HTF source tank fitted at the top of the TESS through a pipe which will be dealt with in the boundary condition section. After transferring heat to the PW mixed with carboxyl functionalized graphene (advanced PCM) kept between the heat exchanger and the stainless-steel cylinder the HTF comes out from the heat exchanger outlet with reduced temperature and the PCM melts by absorbing energy. There are three conduction layers namely SS pipe/cylinder, insulation and the GI (Galvanized Iron) cover which are set up during solution phase after geometry building and meshing. The details of the dimensions of the complete cylinder along with heat exchanger dimensions used in the computation were shown in table 1 and table 2 of (Nagar et al., 2022).

The figures 4 (a) and 4 (b) show the meshed structure of the PCM cylinder. In figure 4 (a) the normal mesh structure of the PCM cylinder is shown isometrically whereas in figure 4 (b) the inside (helical heat exchanger) and outside walls of the cylinder were shown as meshed. The mesh showed very good skewness (0.26) and orthogonality of (0.8). The meshing was done tetrahedrally. The computational

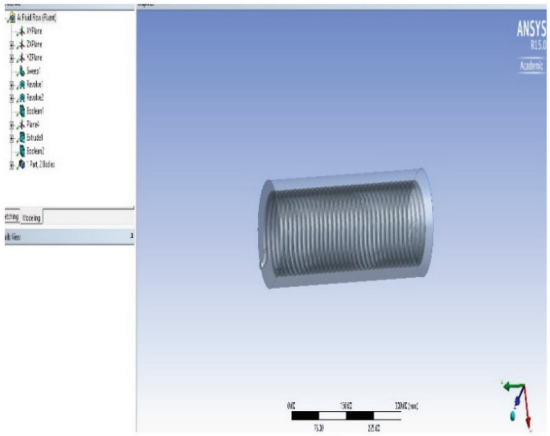


Figure 3(a). Schematic of the PCM cylinder with heat exchanger

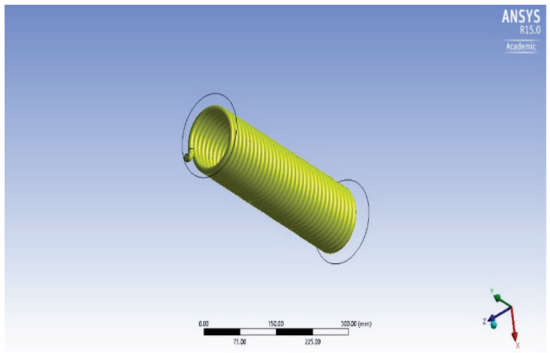


Figure 3(b). Helical heat exchanger

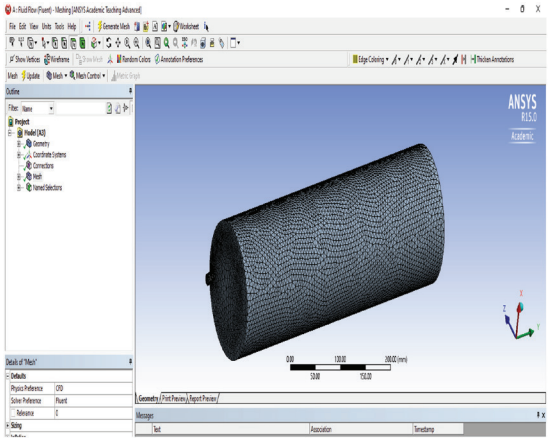


Figure 4(a). Meshed structure of PCM cylinder

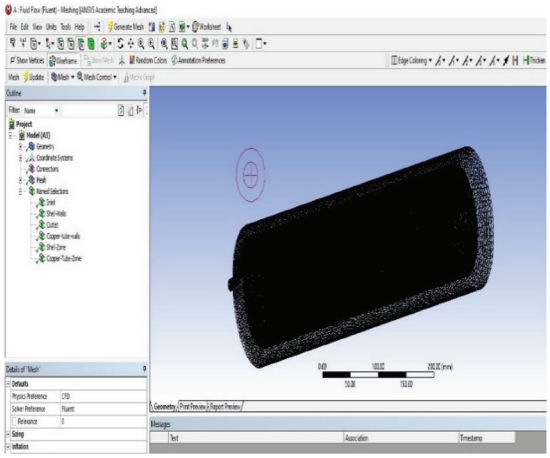


Figure 4(b). The total mesh from inside and outside

domain was represented by a three-dimensional base grid, which had 80,460 mesh cells, with 38,560 cells in the PCM domain and 32,770 cells in the HTF domain. A finer grid was created by increasing the mesh density near the heat exchanger. Grid independence was studied using grid sizes of 0.3 mm, 0.4 mm and 0.5 mm, which resulted in 97,332, 79,468 and 60,613 total numbers of cells, respectively. There was little effect of the grid size on the time evolution of the liquid fraction. It was therefore decided to use a grid size of 0.5 mm with 60,613 cells in all of the subsequent simulations.

## 5.2 NUMERICAL SCHEME AND GOVERNING EQUATIONS

This work used Computational Fluid dynamics (CFD) software ANSYS FLUENT 15.0 academic version for numerical analysis and validation of the experimental work. The theoretical fundamental behind this software was Finite Volume Method (Versteeg and Malalasekera, 2007). The governing equations used were continuity, energy and momentum equations for simulating the melting time on ANSYS FLUENT WORKBENCH and comparing this with the time as obtained from physical experiments thereby validating the results. Boussinesq approximation was also taken in to account for addressing the buoyancy force of air due to density difference. The account of turbulency was also considered by $k-\varepsilon$ turbulence model. The phase change problem has continuity, mass and energy equations. The energy equation takes the form as shown in equation (1).

$$\frac{\partial}{\partial t}(\rho H)+\nabla.(\rho \vec{v} H)=\nabla.(k\nabla T)+S_h \quad (1)$$

Where ρ, $H$, $k$, $\vec{v}$, $T$ and $\mathbf{S}_h$ are density, enthalpy, thermal conductivity, velocity vector, temperature and energy source term, respectively. The enthalpy $H$ is the sum of both sensible enthalpy and latent heat enthalpy.

$$H=h+\Delta H \quad (2)$$

According to equation (2), $\Delta H$ is the latent heat enthalpy and $h$ is the sensible enthalpy.

The sensible enthalpy is given by equation (3),

$$h = h_{ref} + \int_{T_{ref}}^{T} c_p dT \tag{3}$$

Here, $h_{ref}$, $T_{ref}$ and $C_p$ stands for enthalpy at reference temperature, reference temperature and specific heat at constant pressure. The latent heat enthalpy ( $\Delta H$ ) is related to liquid fraction during phase change ( $\beta$ ) and specific latent heat ($L$) by the equation,

$$\Delta H = \beta L \tag{4}$$

Where, $\beta$ is given by the following equation:

$$\beta = \begin{cases} 0 & \mathrm{T} \le T_s \\ \dfrac{T - T_s}{T_l - T_s} & \mathrm{T}_s < T < T_l \\ 1 & \mathrm{T} \ge \mathrm{T}_l \end{cases} \tag{5}$$

Here, $T_s$ and $T_l$ are solidification and melting points, respectively and the model is enthalpy porosity model. The phase changing region or the mushy region is treated as porous where porosity equals the liquid fraction.

The source term of the energy equation (1) is as described by equation (6)

$$S_h = \frac{\partial(\rho \Delta H)}{\partial t} + \nabla.(\rho \vec{v} \Delta H) \tag{6}$$

Because of the formation of fluid during phase change the following set of momentum equation are used:

$$\frac{\partial(\rho u)}{\partial t} + \nabla.(\rho \vec{v} u) = \nabla.(\mu \nabla u) - \frac{\partial P}{\partial x} + S_x \tag{7}$$

$$\frac{\partial(\rho u)}{\partial t} + \nabla.(\rho \vec{v} v) = \nabla.(\mu \nabla v) - \frac{\partial P}{\partial y} + S_y + S_b \tag{8}$$

where $u$ and $v$ are x- and y- direction velocity, respectively; P is the pressure and $\mu$ is the viscosity. $S_x, S_y$ and $S_b$ are the source terms for phase change zone in x-direction, y-direction and due to gravitational acceleration in y-direction denoted by the following equations:

$$S_x = \frac{A(1-\beta^2)}{\beta^3 + \varepsilon} u \tag{9}$$

$$S_y = \frac{A(1-\beta^2)}{\beta^3 + \varepsilon} v \tag{10}$$

$$S_b = \frac{\rho g \beta (h - h_{ref})}{c_p} \tag{11}$$

Where g is the gravitational acceleration, $\varepsilon$ is a small number to prevent division by zero and $A$ is a mushy zone constant. To consider turbulence in this model the turbulent sink term needs to be considered as shown by the following term:

$$S_t = \frac{A(1-\beta^2)}{\beta^3 + \varepsilon} \phi \tag{12}$$

where $\phi$ stands for turbulence parameter obtained from turbulent flow model. Because of the complexity of flow field large eddy simulation model (LES) was used.

### 5.3 GENERAL SIMULATION MODEL ASSUMPTIONS

The type of solver taken was pressure based as the fluid was incompressible. The velocity formulation was absolute and simulation was transient as the melting process was for melting of the PCM with respect to time.

### 5.4 NUMERICAL MODEL AND MATERIALS FOR SETTING UP SIMULATION

In the model section of the ANSYS FLUENT 15.0 (academic version) the energy is switched on/ activated and the viscous k - $\varepsilon$ model having two equations were selected which was a realizable model. In the same model near wall treatment standard wall function was activated for setting up the solution. The solidification/melting model was also activated so, as to find out the time taken for melting of 0.25, 0.50, 0.75, 1.0 volume % functionalized OH graphene mixed with paraffin wax which is one of the main objectives of this research by numerical simulation and thereby comparing it with experimental research as mentioned in this technical article.

### 5.5 CELL ZONE AND BOUNDARY CONDITIONS

The cell zone conditions were given differently for the copper tube and the shell. The copper tube zone had material name as water-liquid, as it has water which is the HTF. The stainless-steel shell contains material named as PW surrounding the copper tube inside the galvanized iron cylinder. The heat transfer occurs from copper tube heat exchanger to the PW doped with COOH group functionalized graphene, which melts by absorbing energy and this results in latent heat energy storage.

The boundary condition for the inlet of this system was for the pipe containing hot HTF having inlet temperature as 85° C entering the inlet of the copper tube heat exchanger. The mass flow rate is entered by suitable conversion to kg/s for three different volumetric flow rates as mentioned in the manuscript, which are 6.25 ml/s, 12.5 ml/s and 25 ml/s. The turbulence intensity is kept as 5 % and hydraulic diameter was 0.0115 m and the specification method was taken as intensity and hydraulic diameter. The outlet was

taken as pressure outlet. The three conduction layers were set for dimensions as per the table 1 and table 2 of (Nagar et al., 2022). The three layers set were the shell (SS pipe), insulation and the GI (galvanized iron) cover. The conduction wall was taken as stationary wall and the copper tube's thickness was fed and simulation was set up and solved.

## 6. RESULTS AND DISCUSSIONS

Herein, the melting time of the advanced PCM along with liquid fraction of the PCM melting is discussed logically.

### 6.1 VALIDATION BY COMPARING EXPERIMENTAL WITH NUMERICAL SIMULATION MODEL FOR MELTING TIME OF ADVANCED PCM (PW WITH COOH GROUP FUNCTIONALIZED GRAPHENE)

The time taken for charging (melting) of different volume % based COOH Group based functionalized graphene doped in PCM (PW) at three different flow rates like 6.25 ml/sec, 12.5 ml/sec and 25 ml/sec of HTF (water) are depicted in figure 5(a), figure 5(b) and figure 5(c), along with their numerically simulated melting times respectively.

By comparing the experimental melting time with numerical simulation time, errors of the first case where flow rate of the HTF (water) was 6.25 ml/sec (inside the copper tube heat exchanger) for 5 different volumes % starting from pristine PW and going to 1 % volume fraction in steps of 0.25 volume % mixed with COOH group functionalized graphene were 1.25 %, 0.67 %, 2.85%, 2.3 % and 2.5 %, respectively having an average error of 1.91 %. Again, In the second case, for flow rate 12.5 ml/sec of the HTF starting from pristine PW and going to 1 % volume fraction in steps of 0.25 % of COOH group functionalized graphene the errors were 1.33 %, 2.14 %, 0.67 %, 2.5 % and 3.63 % with an average error of 2.054 %. Similarly, in third case where flow rate of HTF was 25 ml/sec the errors found out for pristine PW to 1 % volume fraction of the COOH group functionalized graphene mixture (graphene+ PCM) in steps of 0.25 % were 2%, 1.81 %, 3 %, 2.5 % and 3.75 % with an average error of 2.612 %.

The slight difference between experimental and numerical simulation was due to numerical assumptions as stated earlier as well as experimental conditions for performing the experiments. The air gap between the Cu heat exchanger and the SS shell which is filled by PW PCM mixed with COOH based group graphene also contributes to this marginal error. Although the thermocouples used for measuring temperatures were of good quality but there is also scope for measurement errors. The fluctuation in environmental condition at the start of winter season during which experiment was conducted also can be a potential reason for this very small deviation between experimental

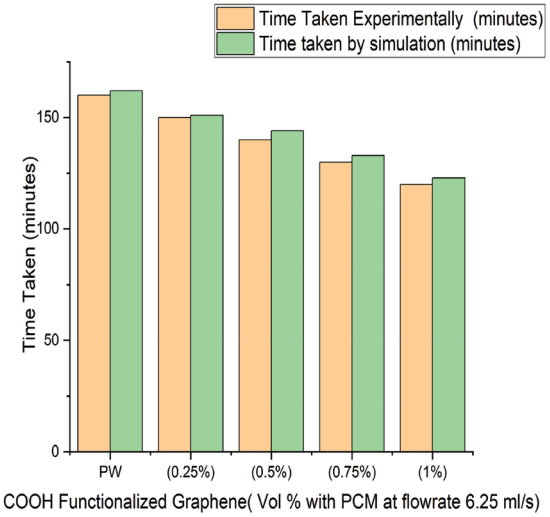



Figure 5(a). Melting time at flow rate 6.25 ml/sec

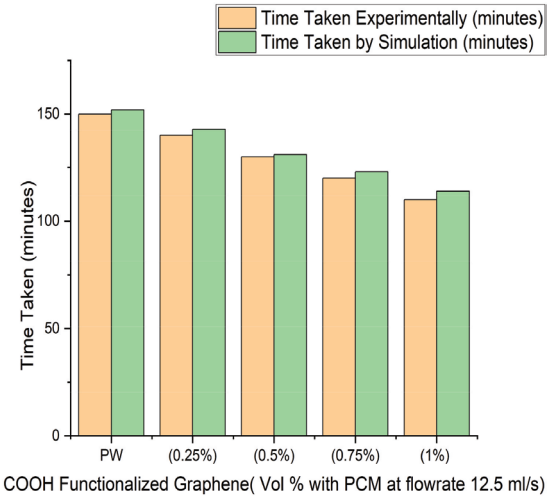



Figure 5(b). Melting time at flow rate 12.5 ml/sec

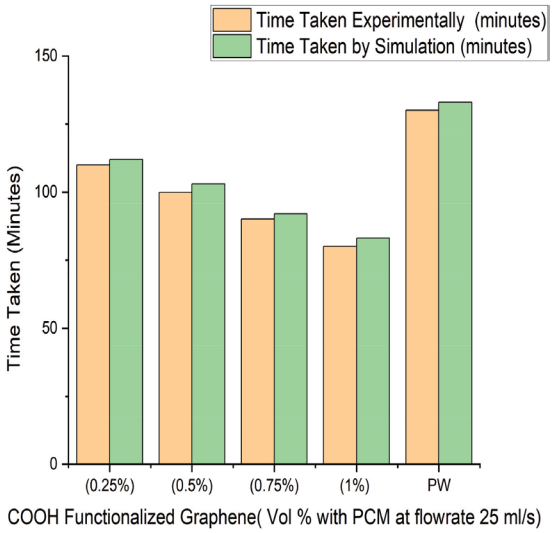
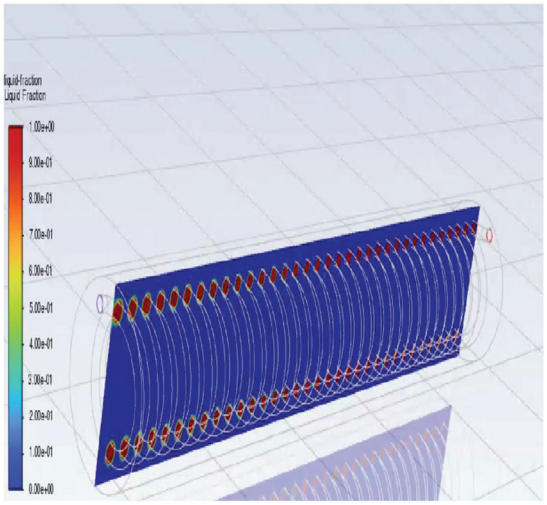
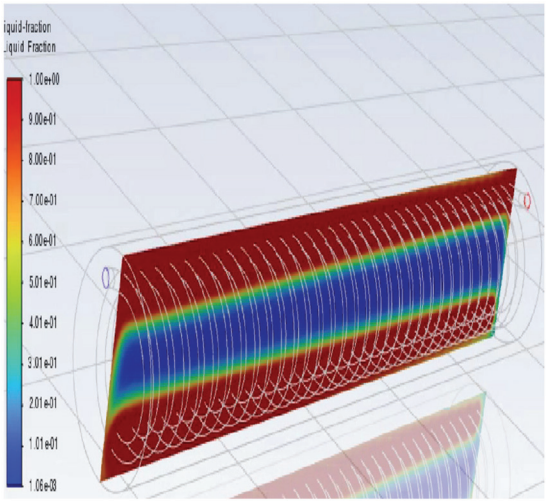



Figure 5(c). Melting time at flow rate 25 ml/sec




(a)

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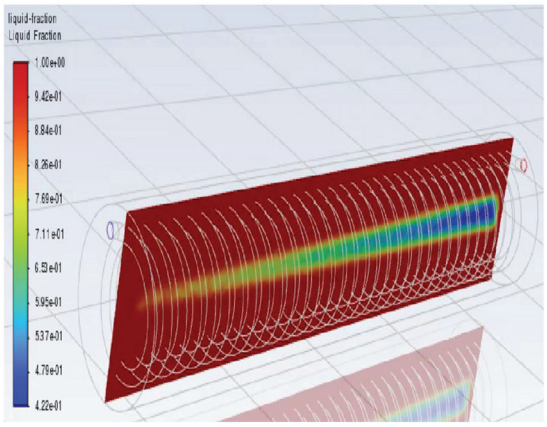
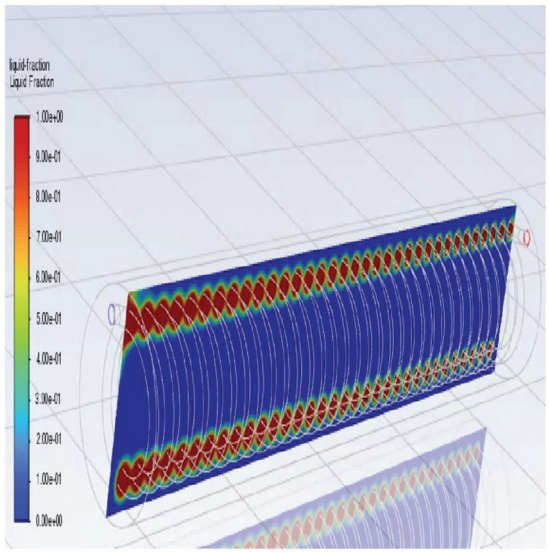
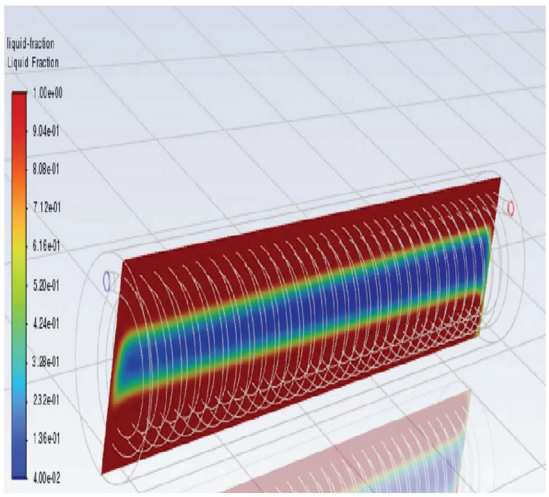



(c)

Figure 6. Liquid Fraction of Pristine PW (a) 10 minutes (b) 60 minutes (c) 130 minutes




(a)

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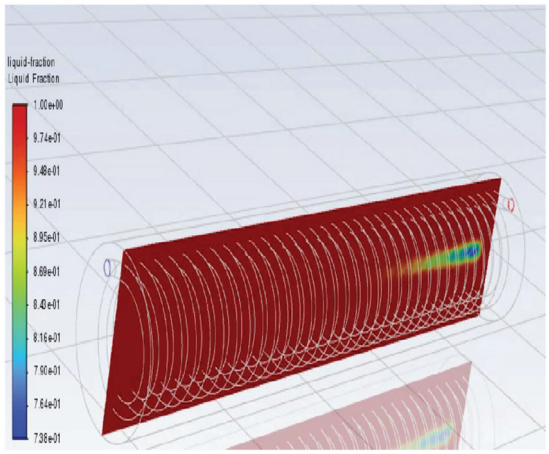



(c)

Figure 7. Liquid Fraction of PW mixed with 0.25 % functionalized graphene (a) 10 minutes (b) 60 minutes (c) 110 minutes

and numerical results. Furthermore, in numerical simulation heat was transferred due to pure conduction by the layers of the concentric cylinders whereas, in actual experimentation condition natural convection happens and this contributes to the mild deviation between numerical and experimental results.

## 6.2 LIQUID FRACTION PROFILES DURING DIFFERENT TIME OF MELTING

The transition of the phase of a body from solid to liquid phase is defined as melting. The main reason of this happening is increment in the internal energy of the system. In the TESS as the warm water goes inside the Cu tube heat exchanger at the fixed temperature of 85°C, it transfers heat to the PW mixed with COOH group functionalized graphene surrounding the heat exchanger inside the stainless-steel shell which causes the PCM to melt thereby absorbing energy.

The liquid fraction profile at various time intervals of melting for PW mixed with COOH group functionalized graphene at a ratio of 0 (pristine PW), 0.25, 0.5, 0.75 and 1 volume % for flow rate 25 ml/sec of the HTF are shown in figure 6, 7, 8, 9 and 10. The liquid fraction is present as a scale (from 0 to 1) where red colour denotes the entire PCM mixed with functionalized graphene has melted, whereas the blue colour indicates the entire advanced PCM is in the solid state. The colour in between blue and red denotes the various volume fraction from 0 to 1. The various frames as obtained through video images of melting at different time intervals were depicted as mentioned below.

The time taken to melt for basic PW is 133 minutes at a flow rate of 25 ml/sec of the HTF. The state of melting by considering the liquid fractions are as shown in figure 6 for various time intervals 10 minutes, 60 minutes and 130 minutes.

The time taken to melt PW mixed with 0.25 % functionalized graphene was 112 minutes at a flow rate of 25 ml/sec of the HTF. The state of melting by considering the liquid fractions are as shown in figure 7 for various time intervals 10 minutes, 60 minutes and 110 minutes.

The time taken to melt PW mixed with 0.5 % functionalized graphene was 103 minutes at a flow rate of 25 ml/sec of the HTF. The state of melting by considering the liquid fractions are as shown in figure 8 for various time intervals 10 minutes, 55 minutes and 100 minutes.

The time taken to melt PW mixed with 0.75 % functionalized graphene was 92 minutes at a flow rate of 25 ml/sec of the HTF. The state of melting by considering the liquid fractions are as shown in figure 9 for various time intervals 10 minutes, 50 minutes and 90 minutes.

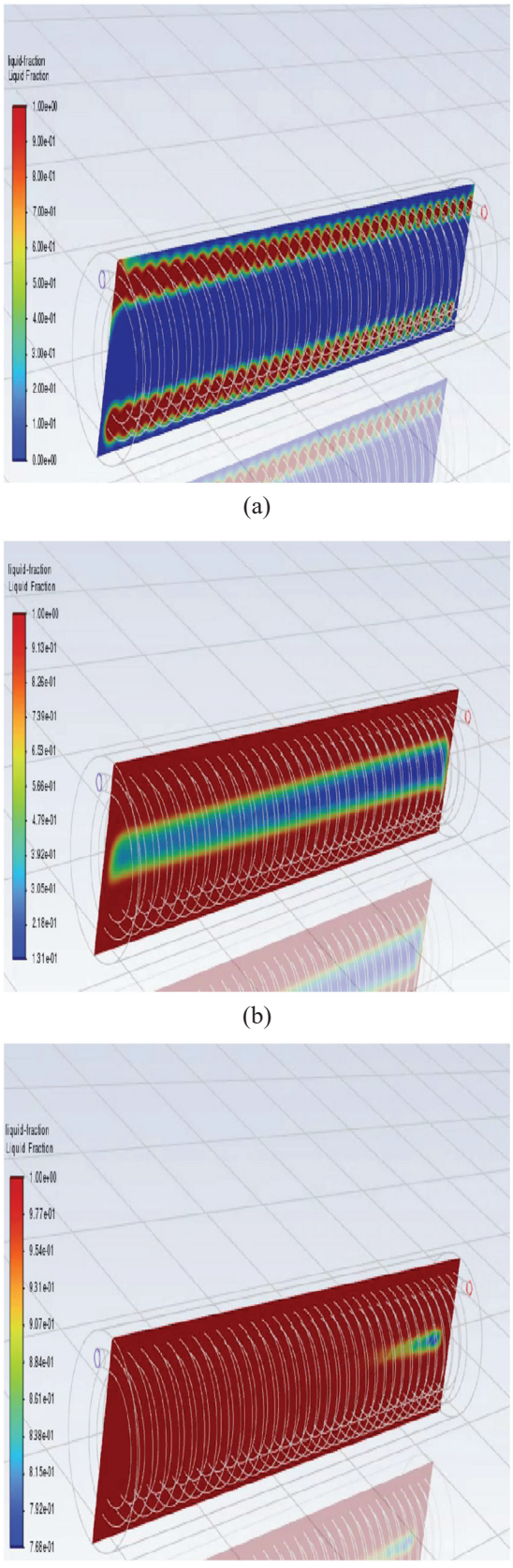
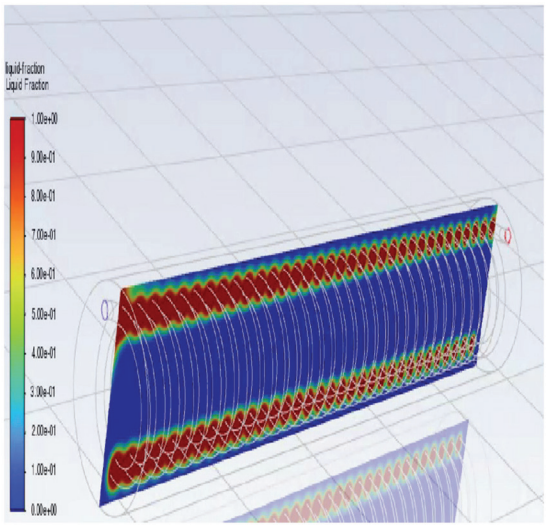
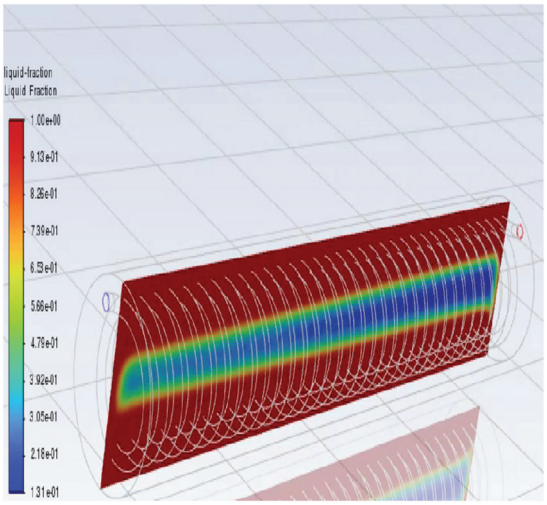


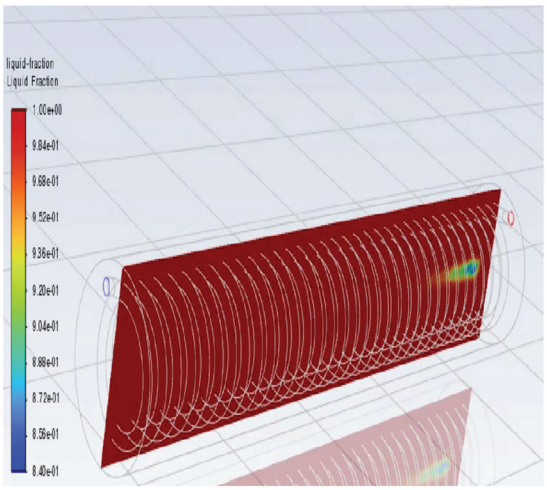

Figure 8. Liquid Fraction of PW mixed with 0.5 % functionalized graphene (a) 10 minutes (b) 55 minutes (c) 100 minutes




(a)

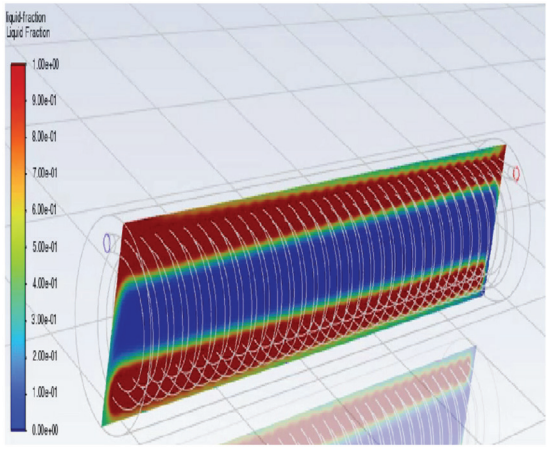



(b)

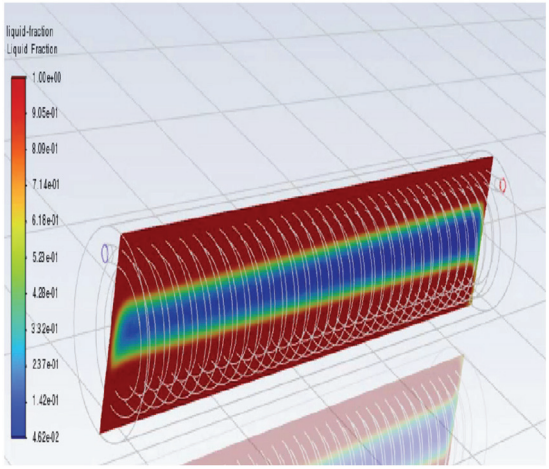



(c)

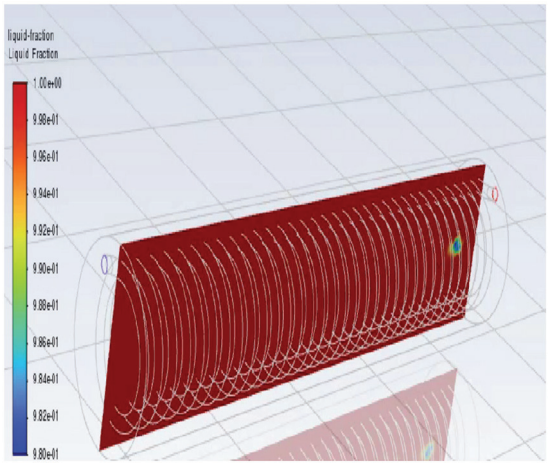
Figure 9. Liquid Fraction of PW mixed with 0.75 % functionalized graphene (a) 10 minutes (b) 50 minutes (c) 90 minutes




(a)




(b)




(c)

Figure 10. Liquid Fraction of PW mixed with 1 % functionalized graphene (a) 10 minutes (b) 40 minutes (c) 83 minutes

The time taken to melt PW mixed with 1 % functionalized graphene was 83 minutes at a flow rate of 25 ml/sec of the HTF. The state of melting by considering the liquid fractions are as shown in figure 10 for various time intervals 10 minutes, 40 minutes and 83 minutes.

## 7. CONCLUSIONS

This present work demonstrates the melting time of the Paraffin wax PCM mixed with COOH group functionalized graphene having different volume fractions like 0.25 %,0.5 %, 0.75 % and 1 % to counter the poor thermal conductivity of the PW PCM at three different flow rates (6.25 ml/sec, 12.5 ml/sec and 25 ml/sec) of HTF which is water. The physical experiments conducted on the thermal energy storage system (TESS) were verified numerically with simulation on ANSYS Workbench 15.0 academic version software. The reported errors were also minimal between experimental and numerically simulated results and were of the average magnitude of 1.91 %, 2.054 % and 2.612% for the three different rates of HTF that are 6.25 ml/sec, 12.5 ml/sec and 25 ml/sec. The melting process was also analysed as defined by liquid fraction profiles stated in the manuscript pictorially. This research also solidifies the fact that as the concentration of COOH group functionalized graphene increases in PW PCM the charging/melting time decreases.

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