From waste chitosan to hybridized scheme thermal energy storage facility: numerical process optimization

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Abstract. Highlighting the utilization of waste material into a value-added fortune is a sustainable win-win solution. Also, the se of solar energy to avoid the energy crisis is essential. In this regard, solar energy storage using the phase **change** material (PCM) at the off-sun periods is a solution. Chitosan augmented with magnetite (ChM) is introduced as a filler for the paraffin's wax (P-Wax) to be a thermal energy storage system. The prepared material is investigated and characterized via X-ray diffractometer (XRD) and Scanning Electron Microscope; SEM Micrographs that exposed the PCM is well prepared. The ChMP is subjected to the PCM system in various mass fractions of 0.1, 0.3, 0.5 and 1.0% to the P-Wax to 0.1-ChMP-Wax, 0.3-ChMP-Wax, 0.5-ChMP-Wax and 1.0-ChMP-Wax. Afterwards, the thermal system is presented and compared with the pristine P-Wax system. The experimental data revealed that the 0.3-ChMP-Wax system showed the pronounced effect reached to 60 kJ stored heat capacity compared to 7 kJ of the pristine system. Thereby, the ChMP-Wax system is an energy storage enhancement system, which might be used in heating application systems. Furthermore, ChMP-Wax system PCM variables are optimized through the application of Box-Behnken design that are corresponding to 70.1ºC of charging temperature, 0.37% ChM addition to the P-Wax and 1.9 g/s of the water flow rate.

1. Introduction

Today, due to the modern lifestyle, one of the major concerns is the energy crisis [1]. Thus, the worldwide energy demand and consumption is in a numerous elevation. Worldwide consumption is associated with the rapid development in industry and domestic life that resulting in a depletion of fossil fuel with severe environmental deteriorations [2-4]. Such impacts are including the toxic and harmful contaminants from the use of traditional fuel. In this regard, engineers, chemists and industrial sectors are working for a solution for such problems [5, 6]. The so-called renewable energy storage is a modern reasonable solution to solve such challenges [7-9]. Renewable energy is required to simultaneously deduce the expensive power generation expenses and eliminate their hazards as a safe solution. The application of renewable energy sources in varied sectors paves the way for economic energy uses Solar energy is categorized as one of a viable candidate among the available renewable energy sources to fulfil all human requirements [10-12]. However, on the other hand, its accessibility might be diluted and intermittent [13]. Consequently, to fulfil the energy demands, solar energy storage is essential. Solar energy storage is storing the sun during the daytime, whereas the sun radiance is available and then to be available at later time when is needed. Thermal solar energy storage is one of forms of the economic simple systems [14-16]. According to the research articles cited [17-22], numerous techniques have been applied for thermal energy storage, while the numerous applied technologies is the phase change materials (PCMs) facility due to simplicity with great solar heat storing capacity [23, 24].

Phase change materials (PCMs) [25] are signified as the substances that could absorb and releasing their latent heat by a change in their physical state [26], namely melting and freezing states [6, 27, 28]. New materials are introduced as a suitable candidate as a phase change materials stored capacity and utilized in a broad spectrum of applications [29-32]. Such materials could be applied in broad spectrum including water heating and district heating, food and pharmaceuticals drying and chemical industry [33-36]. Also, such PCM techniques could supply with partial or full energy supply requirements [37]. The main goal of this study is to introduce a novel PCM approach based on waste stream as a win-win facility. The feasibility of combining chitosan/magnetite nanoparticles for augmented merits with the paraffin PCM stream through the charging and discharging cycles are investigated and compared with the pristine PCM system.

2. Experimental Investigation

2.1. Synthesis of PCM:

Paraffin wax (P-Wax) that owns a melting point of 54°C and an latent heat of fusion of about 190 kJ/kg is applied as the base organic phase change material. Chitosan/magnetite nanoparticles composite material is prepared by simple co-precipitation technique followed by hydrothermal treatment. Primarily, chitosan is dissolved in droplets of acetic acid then is dissolved in 50 mL of distilled water. At the meantime, molar ratios of ferric ($FeCl3$) and ferrous ($FeSO4 \cdot 7H2O$) solutions are dissolved in distilled water. Subsequently, such three solutions were mixed together to achieve a mixture chitosan/magnetite while sodium hydroxide solution is then added to elevate the pH to about 11. Afterwards, the solution was exposed to stirring through heating at 90 °C to attain a precipitate which is then collected. The precipitate is washed successive times with distilled water to reach to a neutral pH followed by vacuum drying at 70 °C.

Additionally, to attain chitosan/magnetite/P-Wax composite with different mass ratios different proportions from the chitosan/magnetite is mixed in a ratio of 0.1, 0.3, 0.5 and 1.0% to the P-Wax to 0.1-ChMP-Wax, 0.3-ChMP-Wax, 0.5-ChMP-Wax and 1.0-ChMP-Wax. To expose a well dispersion of nanoparticles with the P-Wax, the substance is melted and mixed at 60°C through ultrasonic dispersion.

2.2. Methodology:

2.2.1. Experimental configuration:

Experimental indoor and pilot scale outdoor set-up is constructed to check the experimental investigation then reach to real life applications. In such concept, insulated tubular metal box is constructed from galvanized steel and mounted with serpentine type tubes that are fixed on the inner plate of the collector. Also, for the object of trapping the solar radiation inside the box, the flat plate collector is covered with a glass sheet. Such collector is then connected with double pipe heat exchanger. The PCM is filled the inner pipe for melting/solidification processes. The collector heats up the water in the outer pipe of the heat exchanger as the heat transfer to melt the inner pipe PCM material that is called charging cycle. Subsequently, the second cycle is the cooling PCM cycle that is named discharging cycle. The solar energy is stored in PC is then restored through the discharging cycle in a form of hot water in an insulated container. All experiments were carried out around the solar noon during the summer periods. The graphical scheme representing the experimental facility is displayed in Fig. 1. For the object of analysis, digital-type thermocouples are mounted in different places to record the transient temperature values including the PCM charging/discharging cycles and inlet and outlet heat transfer fluid as well as the stored hot water in the insulated container. Also, Eppley Black and White solar-meter type is used to record the solar radiation.

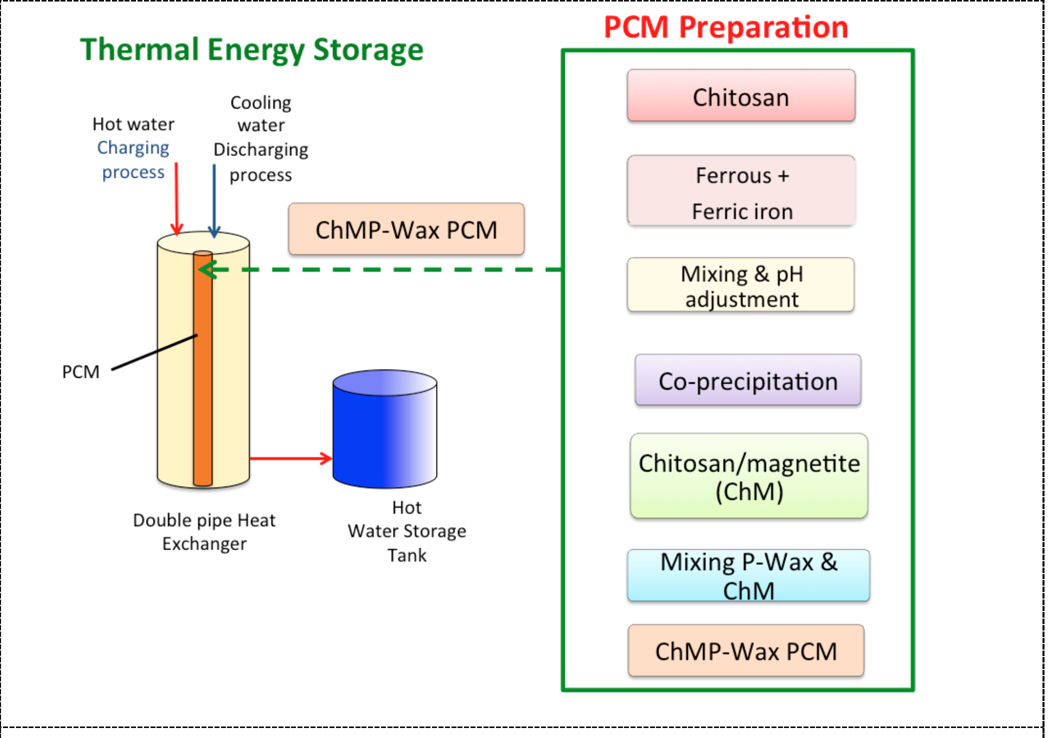

Figure 1. Schematic illustration of the thermal energy storage facility based on ChMP-Wax PCM.

2.2.2. Geographical location of the outdoor facility:
The experimental set-up is mounted under the meteorological conditions in Menoufia Governorate at the north of Egypt whereas the experiments are conducted at Advanced Materials/Solar Energy and Environmental Sustainability (*AMSEES*) Lab., Faculty of Engineering, Menoufia University at the latitude (30°58’N) and longitude (31°01’ E) of the location. The city is endowed with solar radiation and possesses a abundant sunshine through the year especially at the summer season.

2.3. Box-Behnken multi-objective design:
Box-Behnken design model [38] that is based on Response Surface Methodological analysis (RSM) is selected to optimize the operating conditions in order to maximizing the energy storage capacity. In this regard, numerical discharging cycle optimization of the ChMP-wax PCM system is assessed. The most significant variables on the PCM is evaluated and exposed for optimization for the objective of enhance the overall PCM system. RSM as been introduced to deliver numerical assessment of the ChMP-wax system performance through the discharging cycle and optimize the most effectual independent parameters assisted in improving the storage unit. RSM technique that is involving of mathematical methods is used for the multi-variables problems thereby is controlling the process response. The technique is mainly associated with distinctive the system response regarding the appropriate cases. Thus, the current study is based on maximizing the energy storage performance via the main 3-geometry parameters, i.e. charging cycle temperature (T_c); ChMP-Wax portion (%) and the water flow rate that is signified as the system heat transfer carrier. The parameters are identified in their un-codified and codified levels as tabulated in Table 1.

Table 1 Independent parameters with their codified and un-codified levels

Parameters	Symbol		Levels		
			Minimum level	Medium level	Maximum level
	Un-Codified	Codified	-1	0	1
Tc (°C)	E_1	ε_1	68	70	72
ChMP-Wax (%)	E_2	ε_2	0.1	0.3	0.5
$\dot{w}$ (g/s)	E_3	ε_3	1.60	1.80	2.00

Regarding the suggestive model using SAS software, 15-proposed experimental set-up and are tabulated in Table 2 (SAS et al. 1990). In such runs, the minimum, maximum and medium levels of each parameter are listed, but, some runs are repeated for emphasis. Then, the response is recorded through experimental runs and thereby compared with predicted values from the Box-Behnken Design optimization and achieve the maximum value of energy storage capacity. Then, the experimental results are analyzed via the second-order polynomial model equation to fit the response surface regression model (Eq. 1). Furthermore, the heat storing capacity response with the interacting parameters is graphically represented though the use of MATLAB (7.11.0 version) software. Finally, Mathematica software (V 5.2) is used to locate the numerical accurate optimized value of the parameters.

$$\zeta = \beta_o + \Sigma\beta_o\varepsilon_i + \Sigma\beta_{ii}\varepsilon_i^2 + \Sigma\Sigma\beta_{ij}\varepsilon_i\varepsilon_j \quad (1)$$

ANOVA test, the analysis of variance, is performed with SAS (1990) software to statistically assess the system significance.

Table 2. Box-Behnken design in codified and their corresponding un-codified parameters

Run No.	**Codified parameters**			**Un-Codified parameters**		
	ε_1	ε_2	ε_3	E_1	E_2	E_3
1	-1	-1	0	68	0.1	1.8
2	-1	1	0	68	0.5	1.8
3	1	-1	0	72	0.1	1.8
4	1	1	0	72	0.5	1.8
5	0	-1	-1	70	0.1	1.6
6	0	-1	1	70	0.1	2.0
7	0	1	-1	70	0.5	1.6
8	0	1	1	70	0.5	2.0
9	-1	0	-1	68	0.3	1.6
10	1	0	-1	72	0.3	1.6
11	-1	0	1	68	0.3	2.0
12	1	0	1	72	0.3	2.0
13	0	0	0	70	0.3	1.8
14	0	0	0	70	0.3	1.8
15	0	0	0	70	0.3	1.8

3. Results and Discussions:

3.1. Structure and surface morphology of PCM:

The X-ray diffraction (XRD) tests for PCM system is achieved via the use of XRPhillips X'pert (MPD3040) X-ray diffractometer supported by a monochromatic source CuKa (k=1.5406 °A). The XRD measurement is assessed under step-scan mode at a registered diffracted X-rays intensities each 0.02° at a step of 2θ range of 10-60°. Fig. 2 exhibits the XRD pattern for ChMP-Wax PCM material.

It is clear from the XRD pattern in Fig. 2 graph that the spinel structure The main phases of the chitosan and iron oxide exposed as a single face-cantered cubic (fcc) is attained. The existence of the intensive peaks at 2θ of 20.14, 30.52◦, 35.52, 40.41 and 62.84 and are corresponding to [111], [220], [311], [422] and [440], respectively [2]. Such plans are representing the standard magnetite peaks that confirm the presence of magnetite in the sample. Furthermore, according to the pattern, the 2θ of 34.7◦ and 20.14◦ reveals the presence of cellulose material [1]. Besides, the XRD graph verifies the presence of chitosan (Ch) due to the presence of a diffraction peak located at a diffraction angle position 2θ of ~19°. This verifies that the chitosan macromolecules that are augmented to magnetite PCM material.

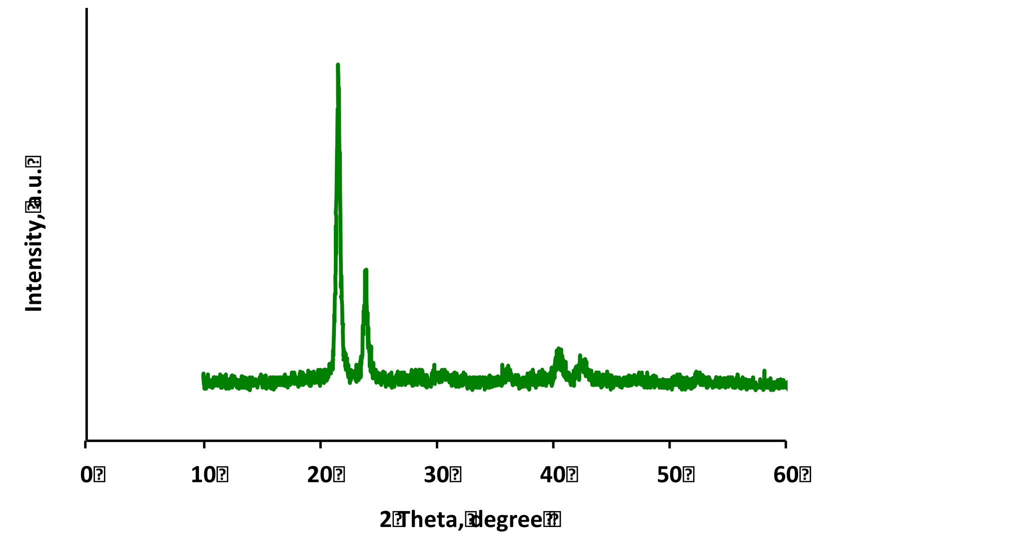

Figure 2. X-ray diffraction patterns of ChMP-Wax composite PCM

Also, the morphology of the synthesized and prepared substance is then checked via the use of scanning electron microscope (SEM) model Quanta FEJ20. The SEM images of ChMP-Wax PCM substance show agglomerated nanoparticles on the surface of wax and represented in Fig. 3. Chitosan cross linker plays a significant role to establish better spherical morphology of nanoparticles. Spherical-shaped particles are representing the magnetite nanoparticles.

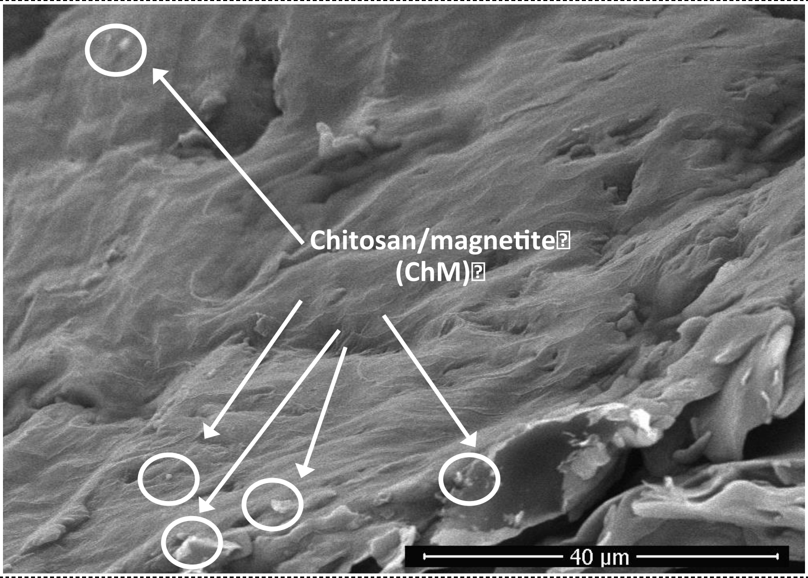

Figure 3. SEM images of ChMP-Wax PCM composite

3.2. Thermal energy storage performance:

3.2.1. Charging (Melting)/Discharging (solidification) cycles:

Melting and solidification cycles are the cycles used for the energy storage and the rate of heat release of the phase transformation materials. The melting and solidification cycles depending on the temperature difference between water as the heat transfer carrier and the phase change material [39]. Consequently, the melting process as the charging cycle and the solidification as the discharging cycle temperatures are dependent on the alteration of the PCM are evaluated and the results are displayed in Fig. 4 (a and b).

The data investigated for the ChMP-Wax PCM composite with respect to time period of melting and solidification at different times re compared to corresponding pristine P-Wax PCM system. Chitosan/magnetite conjugates paraffin wax at different percentages, i.e. 0.1, 0.3, 0.5 and 1.0% to the P-Wax to 0.1-ChMP-Wax, 0.3-ChMP-Wax, 0.5-ChMP-Wax and 1.0-ChMP-Wax are inserted into the base P-Wax paraffin PCM. Obviously, it is illustrated from Fig. 4 (a) ChM conjugates paraffin PCM addition in different proportions outcomes of a significant upsurge of melting temperatures.

The paraffin PCM increase to base P-Wax PCM noted an elevation in the melting temperature. The highest system was noted for 0.3% of 0.3-ChMP-Wax system. The charging cycle temperature increased to reach to 64ºC compared to 52.5ºC for the solo system with no addition to the PCM. But, further increase in ChM percent to the P-Wax results in a decline in the system efficiency. This could be illustrated by the material addition into the base paraffin PCM supports a promising variation in the shape of the heat flow of the PCM. Consequently, the addition of such material modifies and upsurges the value of the melting temperature of such phase change material. Furthermore, it could be estimated the Chitosan/magnetite addition into the base pristine P-Wax as a PCM system enhances its latent heat and thereby controls its photo-degradation [40, 41]. This could be more efficient system in the real application field.

Fig. 4 (b) exhibited the results of discharging process for both solo P-Wax paraffin system and ChM composite P-Wax system as a phase change material. According to the results of the experimental runs, an elevation of the melting temperature of the P-Wax system with the increase of the addition of embedded material. Thus, the solidification temperature will extra improved compared to the corresponding of the pure PCM. However, different temperatures were observed with the varied addition of the embedded material. The overall heat stored from the system is improved and increased. Such result is elucidated by scattered researchers in previous work [39-41].

It is noteworthy to mention that excess embedded material to the P-Wax PCM could be leading to an increase in the dynamic viscosity of the PCM. So, the increase in the dynamic viscosity might decline the rate of the heat transfer rate of the material and this will be unfavorable.

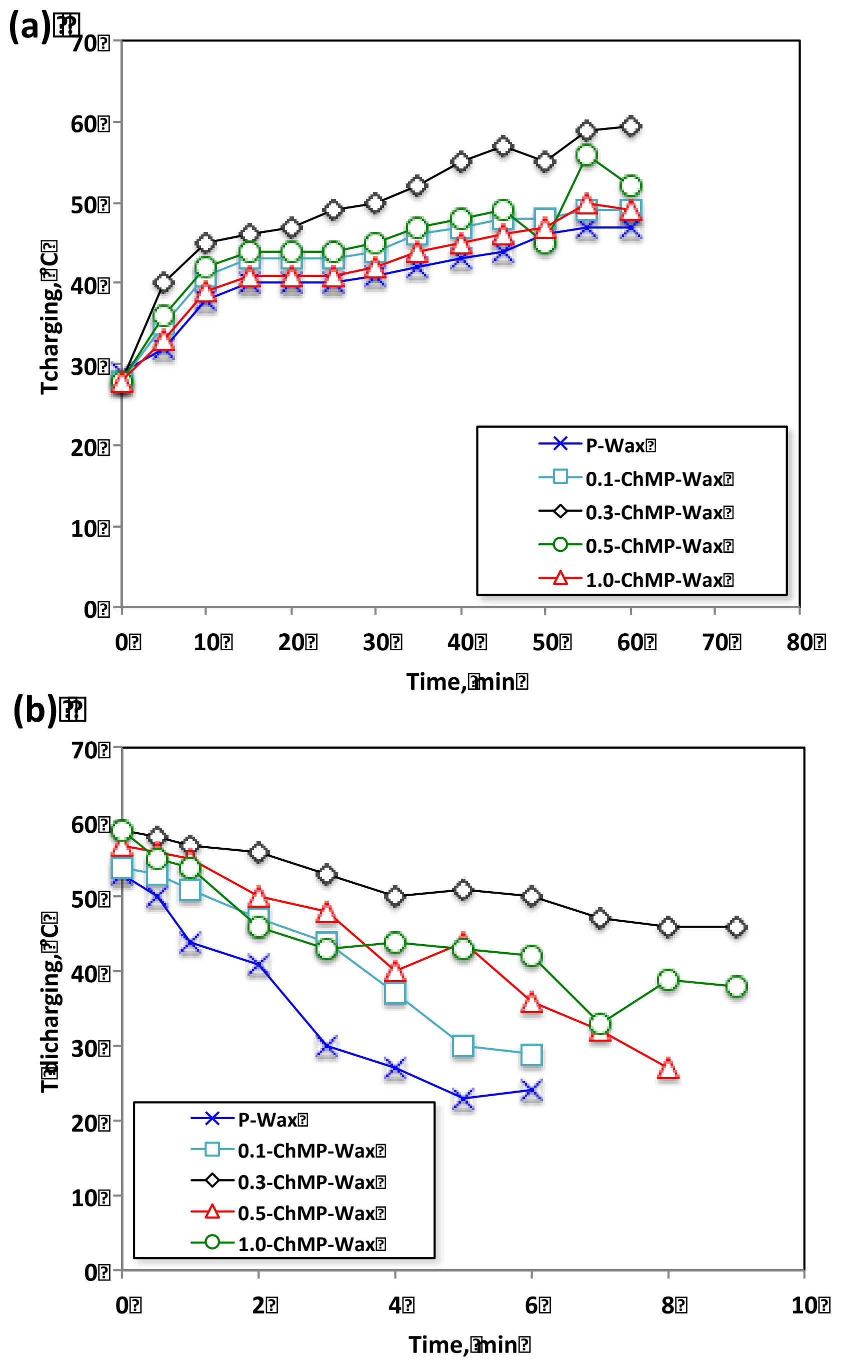

Figure 4. Charging (a) and discharging (b) cycles for PCM systems

3.2.2. Heat stored. Overall heat gained from the process is compared for all the studied the PCM systems and the results is displayed in Fig. 5. The results demonstrated in the figure expose the heat transferred by water as the heat transfer fluid is calculated from equation (2), where: $\dot{m}$: mass flow rate of water (g/s); T: inlet and outlet water temperature difference of inlet and outlet heat exchanger ad C: Specific heat capacity of water (4.18 kJ/kg K) [40]:

$$f = \dot{m}\, C\, T_w \quad (2)$$

The overall heat attained from the PCM substance is calculated through equation (3).

$$Q = mC_p(T_{PCMi} - T_{PCMo}) + m\,HQ_{PCM} = mC_p(T_{PCMi}\text{-}T_{PCMo}) + mH \quad (3)$$

where m is the thermal phase change mass (Kg), *Cp* is the specific heat capacity of PCM (kJ/kg.K), TPCMi and $T_{PCMo}$are temperatures of the PCM of the inlet and outlet double pipe heat exchanger, respectively and H is the latent heat of fusion of PCM (kJ/kg).

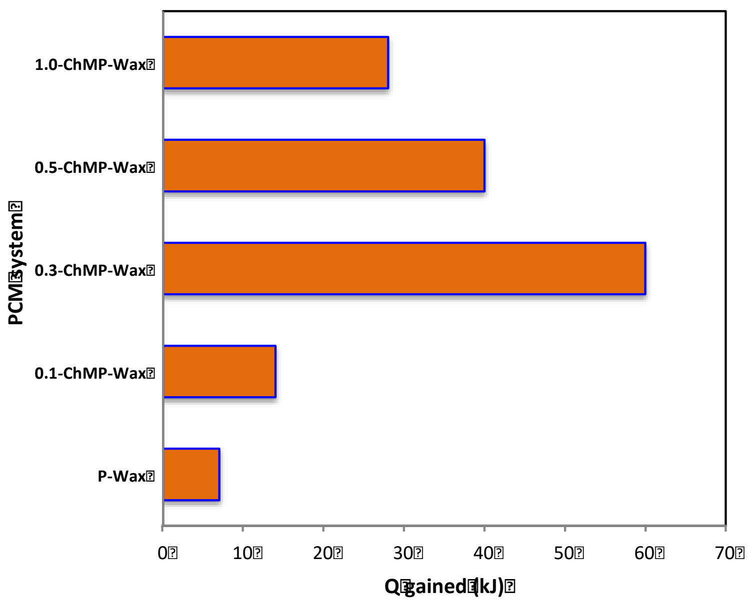

Figure 5. Heat gained through different PCM systems

3.2.3 Box-Behnken optimization strategy. According to the preliminarily results, the optimization criteria is investigated to locate the accurate for the selected most intensive variables optimum operational conditions. The selected levels and the experimental design is displayed In Tables 1 and 2, respectively. The optimization target is to increase the heat gained from the system through the melting temperature as response values. According to the established Box-Behnken design, the experimental experiments were conducted. Afterwards, polynomial model coefficients were processed by the regression equation in terms of coded variables for heat capacity as mentioned as the quadratic model Eq. (4). Also, the numerical predicted optimization attaining a high regression coefficient 0.98 that confirms the goodness-of-fit of the model [38].

$$f = 61 + 1.23E_1 + 1.71E_2 + 10.98E_3 - 10.02E_1^2 - 0.29E_1E_2 + 0.61E_1E_3 - 2.52E_2^2 + 0.23E_2E_3 - 8.11E_3^2 \quad (4)$$

Graphical illustration of the coded parameters and their response are plotted to more assess the interactions between such independent variables and their response, The three-dimensional (3-D) response graphs and the 2-dimensional (2-D) contour plots are augmentable plotted in Fig. 6 by the use of quadratic model design (Equation 4). Commonly, the 3-D plots, the deeper of the color of the graph signify the superior value. But, the degree of curvature of a surface is a sign for the degree of overstated on the response since the more circular contour curvature suggests a lesser interaction influence.

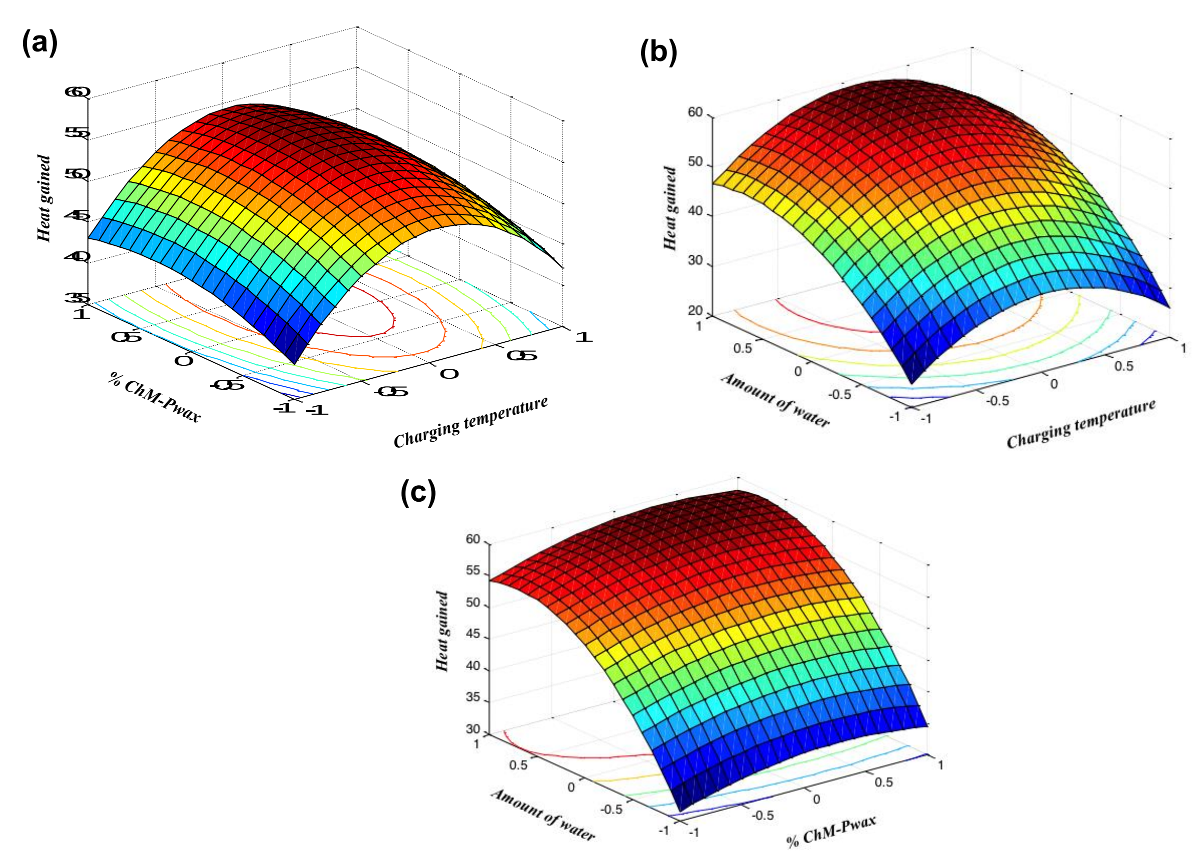

Figure 6. 3D surface plots and their corresponding 2D graphs of heat capacity response (a) Charging temperature and ChMP-Wax weight; (b) Charging temperature and water load and (c) ChMP-Wax weight and water load

In order to validate the numerical model and locate the optimized values of the proposed parameters, Mathematica software (version V 5.2) was applied. The equation of the suggestive quadratic regression model is applied and the statistically proposed values are attained. Next, extra confirmation experiments with three replicates are carried on for verification. Such values are 60 kJ response that are corresponding to 70.1ºC of charging temperature, 0.37% ChM addition to the P-Wax and 1.9 g/s of the water flow rate. The experimental values of the storing efficiency are reached to 61.5 kJ that is so close to the estimated one. Thus, the experimental result also confirms the model prediction.

Conclusion:

Sustainable waste to energy win-win technology has been studied and introduced in the current work. The experimental and numerical study is based on converting the chitosan augmented with magnetite into a value-added material and converting it to a composite to support paraffin wax as a filler for supporting as a phase change material (PCM). Composite embedded paraffin P-Wax PCM showed a pronounced effect on the system reached to na increase in the stored heat capacity from 7 to 60 kJ when 0.3% is added which is corresponding to the optimized use. Furthermore, the system performance is maximized through Box-Behnken optimization that is based on response surface methodology. Hence, such system is introducing win-win technology to attain a sustainable use.

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