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# High density polyethylene spheres with PCM for domestic hot water applications: water tank and laboratory scale study

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## Abstract

Renewable energy is a potential alternative energy provider with fewer CO<sub>2</sub> emissions. However, the mismatch between energy supply and demand is the main disadvantage. Therefore, thermal energy storage becomes an essential technology for enhancing renewable energy efficiency and providing energy supply to the end user. In solar thermal energy systems, hot water tanks are widely used as sensible heat storage technology. Moreover, water storage usually requires large volumes and their improvement has been studied in terms of shape and arrangement. Latent heat storage materials are a potential technology for implementation in water storage tanks in order to reduce their volume and to enhance their efficiency. In this paper, the incorporation of shape high density polyethylene spheres with PCM into domestic hot water tanks is studied. Undesired results obtained in the water tank set-up lead the authors to analyse the PCM leakage in the laboratory. Laboratory analysis pointed out that the PCM-spheres must be thermally cycled and cleaned before their implementation in real application of domestic hot water in order to stabilize the PCM content inside the PCM-spheres.

**Keywords:** thermal energy storage (TES), water tank, phase change materials (PCM), encapsulation, stabilization

## 1. Introduction

Renewable energy systems are characterized as providing energy with fewer CO<sub>2</sub> emissions than conventional systems (Garg et al. 1985). However, the main drawback

39 of renewable energy is the gap between supply and consumption (Gil et al. 2010).  
40 Therefore, energy storage technologies are an important part of the system to ensure the  
41 energy supply available to the end user. Thus, the energy storage system plays a very  
42 important role to define the energy efficiency of the system (Medrano et al 2010).

43

44 In the case of solar energy, the sun as an energy source can be guaranteed only for few  
45 hours a day and it has variable intensity during daytime (Garg et al. 1985). These facts  
46 require a storage system capable of providing heat during periods of reduced solar  
47 radiation (Kenisarin and Mahkamov 2007). Moreover, the use of thermal energy storage  
48 (TES) technologies has high potential to shift or smooth the peak power demand, as it is  
49 demonstrated by Nkwetta et al. 2014a. Nowadays, heat storage in a hot water tank is the  
50 most widely used system, where the sensible heat is stored in a liquid medium. Many  
51 studies have focused on improving these tanks in terms of shape (Altuntop et al. 2005),  
52 material (Esen and Ayhan 1996), envelope (Fazilati and Alemrajabi 2013) and the tank  
53 arrangement (Andersen et al. 2008).

54

55 A promising energy storage technology is the use and implementation of phase change  
56 materials (PCM) (Zalba et al. 2003). In this case, the latent heat absorbed and released  
57 during the phase change from solid to liquid is used. With these materials a larger  
58 amount of thermal energy can be stored compared to the sensible heat absorbed by the  
59 water (Shukla et al. 2009). In addition, the PCM works within a specific temperature  
60 range (phase change temperature), between 55 °C and 70 °C in the case of domestic hot  
61 water tanks, which allows the design of the system to be related to the desired  
62 application for obtaining the maximum amount of energy (Cabeza et al. 2011).

63

64 The inclusion of PCM to improve the performance of the TES systems have been  
65 studied by improving heat transfer through the application of fins, enhancing thermal  
66 conductivity, application of tube-in-shell TES, and using microencapsulation (Nkwetta  
67 and Haghghat 2014). In a study done by Cabeza et al. (2006), the authors concluded  
68 that it is a very promising technology, because it provides hot water for a longer period  
69 of time. Moreover, Nkwetta et al. (2014b) used a numerical investigation to study the  
70 performance of a domestic hot water tank with integrated PCM. The impact of different  
71 PCM, its amount and location inside the water tank were the principal aspects analysed.  
72 The authors concluded that for practical application, the PCM should be placed at the

73 top of the tank to promote stratification and take advantage of it in order to achieve high  
74 energy storage performance. Moreover, Farid et al. (2004) in their review provided a  
75 vision on PCM encapsulation, concluding that macro-encapsulation offered more  
76 benefits in solar thermal energy storage applications.

77

78 In a domestic heating solar-aided system, Esen et al. (1998) studied two different  
79 designs for a latent heat storage tank. A tank with PCM encapsulated in cylinders,  
80 studied in detail by Esen (2000) in a later article, and a tank filled with PCM which  
81 contained pipes were the HTF flows through. Authors concluded that the PCM melting  
82 time depends not only on the thermo-physical properties of the PCM, but also on the  
83 geometric parameters.

84

85 The geometrical configuration of the PCM encapsulation is an issue that Barba and  
86 Spiga (2003) took into account. The performance of three different cases (slab, cylinder  
87 and sphere) was analysed during the discharging process of a domestic hot water tank.  
88 The authors concluded that the best configuration was represented by small spherical  
89 capsules if a rapid discharge mode was desired. The same conclusion drew Wei et al.  
90 (2005) where different geometric properties of the macro-encapsulation were also  
91 studied.

92

93 The encapsulation methods most used in domestic hot water systems or solar thermal  
94 energy storage applications are the macro-encapsulation (also called core-shell) and  
95 shape-stabilized PCM (Milián et al. 2017). Different materials have been used to  
96 contain the PCM such as acrylics, urea, formaldehyde and silica based polymers, metals  
97 and carbon based composites as graphene and graphite, among others (Milián et al.  
98 2017). Shape stabilized PCM presents some interesting advantages such as high amount  
99 of PCM content can be integrated into the material matrix and thermal reliability over a  
100 long time period (Zhang et al. 2006). In this context authors found a commercial  
101 product, where the PCM is impregnated in a high-density polyethylene in spherical  
102 form. Therefore, the inclusion study of a new spherical product containing phase change  
103 materials in a domestic hot water tank solar-assisted is presented in this paper. Different  
104 amounts of PCM inclusion were studied in order to determine the most appropriate  
105 implementation according to their thermal behaviour. Moreover, a laboratory analysis of  
106 the spherical product was carried out to evaluate its thermal stability.

107 **2. Pilot plant scale**

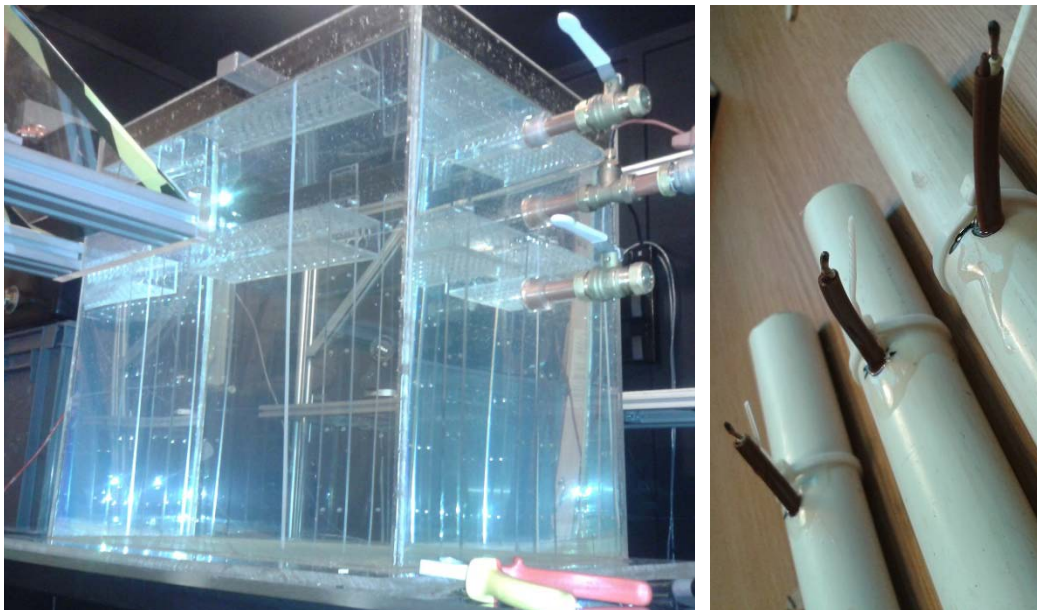
108

109 **2.1. Experimental set-up**

110

111 An experimental set-up was prepared to analyse the effect of adding phase change  
112 materials (PCM) in the top part of a water tank. The experimental set-up consisted of a  
113 transparent acrylic plastic water tank of 600 x 400 x 500 mm supplied by an external  
114 power source that simulated a solar collector. The tank was instrumented with 15  
115 thermocouples type-T prepared in the laboratory according to the EN-60584-1:2013,  
116 related to the applicable accuracy to this type of sensors, with a standard deviation of  
117 0.16 °C. The sensors were placed inside a PVC pipe as shown in Figure 1 in order to  
118 measure the temperature of the water at different levels of the tank.

119



120

121

Figure 1. Left, water tank installation; right, thermocouples type T.

122

123 A commercial product (Ball-ICE® marketed by PCM Products) of polyethylene spheres  
124 impregnated with a paraffin mixture that has a melting point of 58 °C (Figure 2), were  
125 tested. The spheres were analysed with the differential scanning calorimetry (DSC) in  
126 order to obtain their thermal properties and the results are shown in Table 1 together  
127 with the ones provided by the manufacturer.

128

129

130 Table 1. PCM spheres physical properties.

PCM spheres A58	Manufacturer	DSC	
Phase change temperature	58	$T_m$ 55 – $T_s$ 45	(°C)
Density	910	-	(kg/m <sup>3</sup> )
Latent heat capacity	132	135	(kJ/kg)
Volumetric heat capacity	120	-	(MJ/m <sup>3</sup> )
Specific heat capacity	2.2	-	(kJ/kg·K)
Thermal conductivity	0.22	-	(W/m·K)

$T_m$ : melting temperature;  $T_s$ : solidification temperature

131



Figure 2. PCM spheres (Ball-ICE®).

132

133

134

## 135 2.2 Methodology

136

137 The experiments consisted of heating the water tank up to 62 °C. Once the water  
138 reached the maximum temperature these conditions were maintained during 1 hour and  
139 afterwards the tank was cooled down naturally to 32 °C. This experiment was carried  
140 out with the water tank without PCM and with different amounts of PCM spheres, thus  
141 increasing the energy storage capacity of the water tanks by 17% and 33%, respectively  
142 (Table 2). Each experiment was duplicated to have repeatability.

143 The storage density provided in Table 2 is calculated through the addition of the energy  
144 provided by the amount of PCM spheres ( $Q_{pcm}$ ) to the energy provided by the water in  
145 the tank ( $Q_{water}$ ). The following equations were used:

146  $Q_{pcm} = m_{pcm} \cdot L_{pcm}$  (1)

147 where,  $m_{pcm}$  is the total PCM spheres mass incorporated in the system and  $L_{pcm}$  is the  
 148 latent heat of fusion.

149  $Q_{water} = m_{water} \cdot Cp_{water} \cdot \Delta T_{exp}$  (2)

150 where,  $m_{water}$  is total water mass,  $Cp_{water}$  is the water specific heat capacity,  $\Delta T_{exp}$  is the  
 151 temperature difference performed in the water tank during the experiment (62 °C to 32  
 152 °C).

153

154 Table 2. PCM spheres mass added to the water tank.

TES material	Theoretical increase [%]	Storage density [kJ] ( $\Delta T=30$ °C; V=100 L)
Water	-	12544
10.5 kg PCM spheres + water	17	14623
21 kg PCM spheres + water	33	16702

155

156 2.3 Results

157

158 First of all, an experiment without PCM was performed in order to have a reference  
 159 pattern, which will be used to discern the effects of PCM inclusion. The water blank  
 160 experiments took 920 and 932 minutes, respectively, to cool down to 32 °C.

161

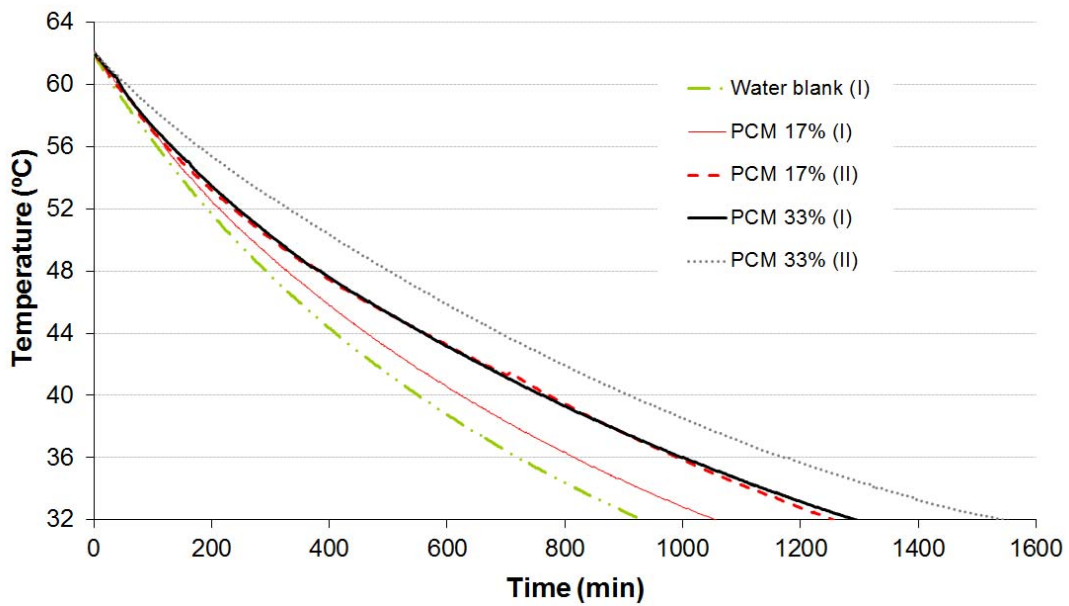
162 The next test increased 17% the storage capacity of the tank with the addition of 10.5 kg  
 163 of PCM spheres. The discharge period was increased compared to the water blanks,  
 164 lasting 1057 min. In the second replication of this experiment the authors found out that  
 165 keeping the tank at 62 °C for a period of about 10 hours after this temperature was  
 166 reached, the cooling down process lasted longer (1256 min). This fact could be  
 167 occurring because in the first test the PCM was not completely melted due to the low  
 168 thermal conductivity of the plastic material of the spheres.

169

170 The same effect was registered when the storage capacity of the tank was increased to  
 171 33% by using 21 kg of PCM spheres. When the charge period was longer the cooling  
 172 rate lasted 1543 min while a shorter charge period corresponds to 1295 min to cool it  
 173 down.

174

175 Figure 3 shows the average temperature of the water tank for the experiments described  
 176 above with different amounts of PCM. The experiments with PCM addition do not  
 177 show the expected flat line around the phase change temperature (58 °C) due to the  
 178 phase change process. Nevertheless, different cooling rates were registered and a delay  
 179 at the end of the discharging process were seen when PCM was added, most probably  
 180 due to the storage effect. A summary of the data obtained in the experiments is shown in  
 181 Table 3.  
 182



183  
 184 Figure 3. Mean bulk temperature in the tank during cooling process in the water blank, PCM  
 185 17% and PCM 33%.

186  
 187 Table 3. Summary of experimental results.

		PCM	Discharge period (min)
<b>Water blank</b>	<b>1</b>	Without	932
	<b>2</b>		920
<b>PCM 17%</b>	<b>1</b>	10.5 kg of PCM	1057
	<b>2</b>		1256
<b>PCM 33%</b>	<b>1</b>	21 kg of PCM	1295
	<b>2</b>		1543

188



189 During the experiments the authors observed that the PCM spheres were losing the  
190 paraffin wax mixture. The leakage was completely visible at the end of the experiments  
191 as Figure 4 shows, the spheres were encapsulated in solidified paraffin. This effect  
192 should be a problem affecting the storage performance of the PCM spheres, as it may  
193 lose storage capacity, and also the system (water pumps, pipes) where the PCM is  
194 implemented could be damaged. Furthermore, any direct storage system may lead to the  
195 PCM being discharged to taps. This phenomenon was also detected in McClenaghan et  
196 al. (2014) study where the wax leaked from the spheres and solidified in the exit pipe  
197 blocking the flow.  
198



199  
200 Figure 4. PCM spheres (Ball-ICE®) after the tests in the experimental set-up  
201

202 Since the authors registered unexpected thermal behaviour in the water tank installation  
203 and phase change material leakage from the spheres, a laboratory methodology was  
204 defined in order to identify the phenomenon occurring with this type of material.  
205

### 206 **3. Laboratory analysis**

#### 207 208 3.1. Methodology 209

210 In order to analyse the PCM lost out of the spheres two laboratory scale methodologies  
211 were designed and evaluated. The aim of these methodologies were to detect the rate of  
212 the PCM leakage and if this loss affected the energy storage capacity of the PCM-  
213 spheres.  
214

215 First of all the PCM-spheres were pre-treated based on the manufacturer  
216 recommendations in order to remove the excess of PCM and to have spheres that were  
217 materially stabilized. The pre-treatment consisted of cycling the samples five times by  
218 heating up and cooling down the spheres. The water was changed after each thermal  
219 cycle, thereby PCM excess is extracted.

220

221 Once the pre-treatment was done to all the samples, two different methodologies were  
222 followed. The first one is named as *Cycling* consisting of:

- 223 1. Submerge the PCM sphere into water the PCM sphere in a glass beaker of 150  
224 mL (see Figure 5).
- 225 2. Heat up the beaker from 30 °C to 70 °C during 3h in a programmed muffle  
226 furnace.
- 227 3. The temperature inside the muffle furnace is maintained at 70 °C for 5:40 h.
- 228 4. Cooling down naturally to 30 °C.
- 229 5. Repeat again from step 1 for 25 thermal cycles.

230



231

232 Figure 5. Submerged PCM sphere in a glass beaker of 150 mL water.

233

234 The second methodology called *Washing* follows the same procedure as that for *Cycling*  
235 but after each thermal cycle the water is changed to perform the next cycle with clean  
236 water.

237

238 Initially, a sample of a PCM-sphere that was not treated with any of those  
239 methodologies (*Cycling and Washing*) was analysed as a starting point result. A further  
240 sample of a PCM-sphere, removed from the experimental water tank installation after  
241 the testing described in section 2, was also analysed.

242

243 Additionally, five PCM-spheres were tested for each methodology (*Cycling* and  
244 *Washing*) 5, 10, 15, 20, and 25 times, respectively. Three triplicates of each samples  
245 was performed. Samples of each sphere as well as the washing/cycling water were  
246 analysed. Thereby, after several cycles the amount of PCM that was remaining inside  
247 the spheres as well as the paraffin flowing out to the washing/cycling water could be  
248 analysed and compared to the initial quantity.

249

250 The quantity of PCM inside the spheres after washing/cycling tests was analysed using  
251 DSC. This is one of the most powerful techniques to analyse the thermophysical  
252 properties of substances as melting temperature and melting enthalpy. The melting  
253 enthalpy was characterized in order to understand the PCM quantity of the spheres.  
254 Thus, the PCM percentage in weight is calculated following Eq. (1):

$$255 \quad \%_{wt(PCM)} = \frac{\Delta H_{sample}}{\Delta H_{purePCM}} \cdot 100 \quad (1)$$

256

257 The DSC analyses were performed under 0.5 K/min heating rate between 30 - 75 °C  
258 under 50 mL/min H<sub>2</sub> flow within 40 ml aluminium crucible with a DSC822e from  
259 Mettler Toledo.

260

261 The paraffin content on the washing/cycling water was also quantified by using gas  
262 chromatography coupled with a mass spectrometer (GC-MS).

263

### 264 3.2. Results

265

266 DSC results are shown in Table 4. Several samples from several parts of each sphere  
267 were evaluated. Authors found out that the spheres provided by the manufacturer (PCM  
268 Products) do not have the same PCM content, hence PCM quantity should not be  
269 directly compared. For this reason, the PCM content of the sample was analysed in the  
270 centre and on the external surface of the sphere and a ratio of difference between these  
271 samples was calculated. Moreover, the initial samples (without any washing-cycling  
272 process) and a sample used in the experimental study at pilot plant scale have been  
273 analysed as well.

274

275 The results are compared in Table 4 where the percentage in weight of PCM is  
 276 calculated based on the DSC results and Eq. (1). Results show that the sample under  
 277 pilot plant conditions, the sample washed 25 cycles and the initial sample have almost  
 278 the same PCM content difference between centre and surface, while the PCM content  
 279 for the sample cycled 25 times presents a higher difference. This is due to the PCM  
 280 migration from the middle of the PCM-spheres to the surface when it is thermally  
 281 cycled.

282

283 Table 4. PCM content of sample (average of three triplicates) under study based on DSC results  
 284 and calculated following Eq. (1)

	<b>Centre (kJ/kg)</b>	<b>Surface (kJ/kg)</b>	<b>(% difference between centre-surface)</b>
Not treated	61.0	56.6	7.2
Washed	61.5	57.2	7.1
Cycled	78.5	59.4	24.3
Experimental water tank	64.7	59.3	8.5

285

286 On the other hand, the GC-MS results have been analysed and the paraffin content into  
 287 the washing/cycling water was quantified. Table 5 lists the paraffin content into the  
 288 water. When PCM spheres are used following the Washed methodology, which  
 289 simulates a direct charge/discharge application, there is no significant PCM leakage;  
 290 when the Cycled methodology is used, which simulates an indirect application, the  
 291 PCM leakage can be substantial and would affect the system components.

292 These results indicate that the PCM-spheres must not just be pre-treated but thermally  
 293 cycled between 20 and 25 times before their use, in order to stabilize the PCM content  
 294 and achieve proper results.

295

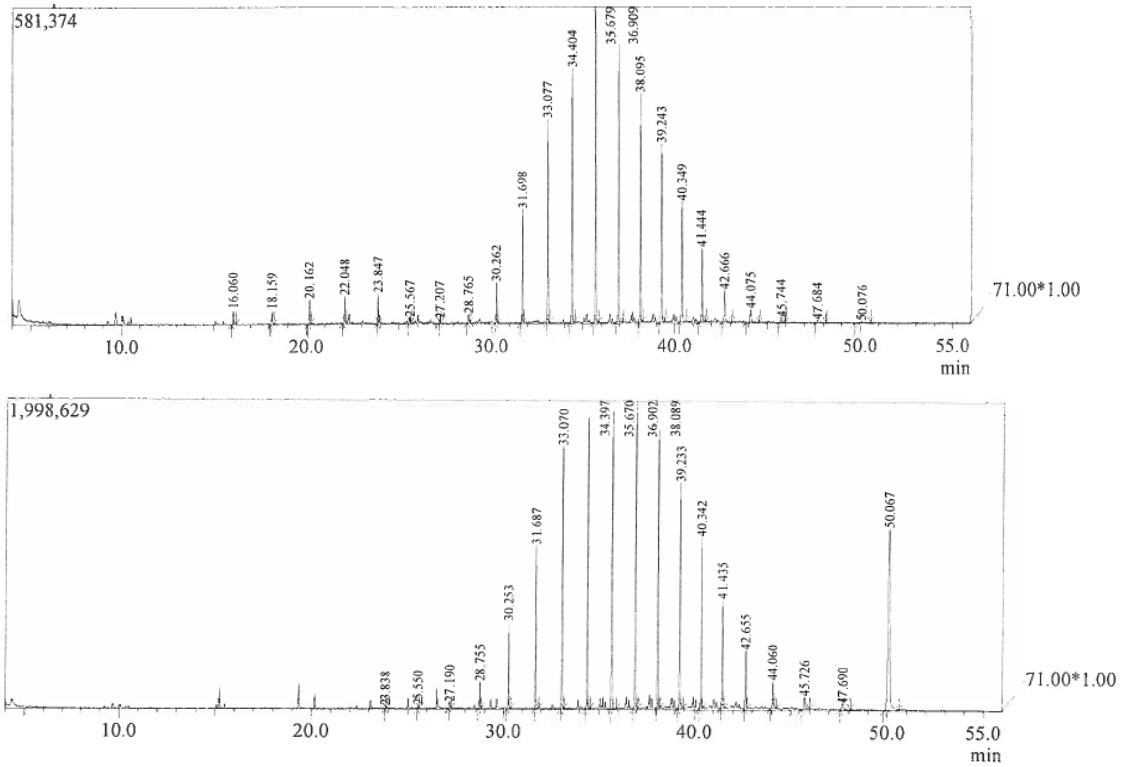
296 Table 5. PCM content into the water used to wash the PCM-spheres.

	<b>5 cycles sphere (mg/kg)</b>	<b>10 cycles sphere (mg/kg)</b>	<b>15 cycles sphere (mg/kg)</b>	<b>20 cycles sphere (mg/kg)</b>	<b>25 cycles sphere (mg/kg)</b>
Washed	0.54	0.15	0.1	1.17	0.23
Cycled	120	170	330	230	255

297

302 Moreover, the chromatograms obtained of the sample washed 25 times and cycled 25  
303 times are presented in Figure 6. Note that the PCM chemical composition of the  
304 washing/cycling water samples is very similar, therefore PCM was not degraded during  
305 testing.

306



307

308

309 Figure 6. Chromatogram of the paraffin wax PCM contained on cleaning water (Cycled samples  
310 on the top, and Washed samples at the bottom).

311

## 312 5. Conclusions

313

314 An experimental set-up consisting of a water tank supplied by a heat source was used to  
315 test the energy density improvement with the implementation of phase change  
316 materials. Polyethylene spheres in which PCM is embedded were located at the top of  
317 the water tank in order to enhance the stratification and hence to improve the energy  
318 density.

319

320 Results obtained in the experimental set-up of the water tank showed that the effect of  
321 PCM is beneficial to keep the water hot for longer. However, ICE-Balls with A58 PCM  
322 are made of polyethylene, which means that they have a low thermal conductivity. This

319 fact makes it difficult to melt the PCM inside the spheres and it takes long time to  
320 charge the PCM which is inside the tank.

321

322 Moreover, PCM leakage was observed during the tests undertaken in the water tank and  
323 a laboratory methodology was designed to analyse this phenomenon since it strongly  
324 affects the use of PCM in a real application.

325 The laboratory analysis indicates that the PCM-spheres must be thermally cycled and  
326 cleaned before their implementation in real application of domestic hot water in order  
327 for the PCM content to be stabilized inside the spheres. Moreover, the paraffin wax-  
328 PCM floating on the system water should be controlled; otherwise thermal performance  
329 efficiency could decrease affecting the charging/discharging system processes.

330

331 There are several recommendations/alternatives to enhance the way to proceed when  
332 PCM-spheres are implemented in domestic hot water system. First of all, at least 25  
333 thermal-cycling process is recommended before the PCM-spheres are introduced. Some  
334 alternatives could be done to improve the thermal performance of the PCM-spheres: to  
335 use a coating on the external surface of the spheres in order to ensure the PCM stability  
336 inside the spheres and to change the material used as the matrix in order to enhance the  
337 thermal conductivity and reduce the PCM leakage.

338

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340

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