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Synthesis of Functional Nanocapsules and their Application to Cotton Fabric for Thermal Management

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24 problems, researchers have been working on smart textiles or intelligent textiles. Phase change
25 materials are active smart materials which can sense and react to the environmental stimuli by
26 storing and releasing heat energy (Onofrei et al. 2010). Phase change materials cannot be applied
27 directly on textiles because they melt by absorbing heat or crystallize by releasing the same heat.
28 To prevent phase change materials from leakage, a process called microencapsulation or
29 nanoencapsulation is used for durability and better application (Mondal 2008).

30 PCMs are attractive for storing energy in all the available heat energy storage techniques due to
31 the high density, compact storage system and high latent heat (Farid et al. 2004; Mondal 2008).
32 The pioneer study of phase change material was applied for space crafts on small scale and then
33 on large scale was applied in buildings and solar energy systems to build thermal energy storage
34 system (Buddhi et al. 1987; Lacroix 1993; Pauken et al. 2007). A large number of inorganic and
35 organic PCMs are available in the temperature range of $-5\text{ }^{\circ}\text{C}$ to $190\text{ }^{\circ}\text{C}$ (Agbossou et al. 2010;
36 Alkan 2006; Choi et al. 2001). The organic phase change materials ranging from $18\text{-}65\text{ }^{\circ}\text{C}$ are used
37 in textiles and buildings to enhance thermal comfort effect (Tyagi and Buddhi 2007; Zalba et al.
38 2003). Among all the PCMs, n-octadecane is usually used for the textile application having melting
39 point of $28\text{ }^{\circ}\text{C}$ (Feldman et al. 1986).

40 Microencapsulation is the process of covering core material such as PCM in a protective shell
41 called capsule. Microcapsules define the particle size in the range of $1\text{ }\mu\text{m}$ to $1000\text{ }\mu\text{m}$ while
42 nanocapsules are used to describe the particle size less than $1\text{ }\mu\text{m}$ (Sarier and Onder 2012). There
43 are many techniques for microencapsulation such as complex coacervation (Uddin et al. 2002) *in-*
44 *situ* polymerization (Jin et al. 2008), interfacial polymerization (Chen et al. 2012), and spray drying
45 (Borreguero et al. 2011). *In-situ* polymerization is widely used among all of these synthesis

46 techniques because of rapid wall formation and also strong capsule formation suitable for textile
47 (Mondal 2008; Zhao and Zhang 2011).

48 **Shin et al. (2005)** synthesized microcapsules containing n-eicosane as core material and melamine
49 formaldehyde as shell material by *in-situ* polymerization. They determined that the latent heat of
50 MPCM was 134.3 Jg^{-1} with particle size of $1.89 \text{ }\mu\text{m}$. The maximum latent heat of thermo-
51 regulating textiles after application was determined as 4.44 Jg^{-1} . **Sarier and Onder (2007)** used urea
52 formaldehyde as shell material and different paraffin; n-hexadecane, n-octadecane and n-eicosane
53 as core materials. The average particle size of microcapsules was found to be $69.1 \text{ }\mu\text{m}$ with 51.7 -
54 54.8 Jg^{-1} of latent heat. **Fang et al. (2010)** prepared microencapsulated phase change materials
55 using silicone dioxide as encapsulating shell and paraffin as core material via Sol-Gel method. The
56 differential scanning calorimetry results showed that the latent heat of microencapsulated paraffin
57 was 165.68 Jg^{-1} . **Li et al. (2011)** encapsulated n-hexadecane using urea formaldehyde as shell
58 material. They studied the effect of surfactant on the process of nanoencapsulation and obtained
59 the average capsule diameter of 270 nm . They determined that as the amount of surfactant
60 increases, the particle size of nanocapsules decreases affecting the thickness of wall of particles;
61 latent heat was found in the range of 114.6 - 143.7 Jg^{-1} .

62 **Salaün et al. (2009)** synthesized microcapsules using melamine formaldehyde as shell material and
63 different paraffin as core material with their binary mixtures. They added 4% wt. tetra ethyl ortho-
64 silicate to enhance latent heat of phase change material. **Sánchez et al. (2010)** microencapsulated
65 paraffin wax from C_{19} to C_{27} sourced commercially as a mixture using polystyrene as shell
66 material. The melting temperature of MPCM was from 40°C to 45°C . They coated 100% cotton
67 fabric with prepared microcapsules using different commercial binders to characterize the
68 thermoregulating effect of the fabric and 7.6 Jg^{-1} of latent heat was found on fabric containing 35%

69 wt. of MPCM. The time duration for thermoregulating effect increases with the increase of the
70 amount of latent heat and hence more Jg^{-1} is required where longer time duration of
71 thermoregulating effect is required.

72 Nanoencapsulation has been paid attention by more researchers now days because of narrow
73 particle size and enhanced surface characteristics when they are applied on textiles for
74 thermoregulating effect (Sarier and Onder 2012).

75 **Sari et al. (2009)** synthesized nanocapsules using PMMA (polymethyl methacrylate) as a shell
76 material and n-octacosane as a core material for thermal energy storage. The melting temperature
77 was measured as $50.6\text{ }^{\circ}\text{C}$ containing latent heat of 86.4 Jg^{-1} . The average diameter of the
78 nanocapsules was $0.25\text{ }\mu\text{m}$. In 2010, **Kwon et al. (2010)** developed NPCM (nanoencapsulated
79 phase change materials) with particle size less than 100 nm. They used polystyrene and PMMA as
80 a shell material to encapsulate n-octadecane as PCM. The latent heat measured for polystyrene
81 and PMMA based nanocapsules were 114 Jg^{-1} and 120 Jg^{-1} respectively. Nanocapsules of n-
82 hexadecane with PMMA shell was made by **Black et al. (2010)** with the particle size between 100
83 nm and 280 nm. **Alay et al. (2010)** studied nanocapsules of PMMA shell containing n-hexadecane
84 as core material with an average diameter of 260 nm and 148.05 Jg^{-1} of latent heat at $17.23\text{ }^{\circ}\text{C}$.
85 They incorporated nanocapsules into the PAN fibre during electrospinning and measured the heat
86 of fusion which was 36.80 Jg^{-1} .

87 More recently **Karthikeyan et al. (2014)** made NPCM containing urea formaldehyde as a shell and
88 paraffin wax as a PCM using *in-situ* polymerization technique; the average particle size was 256
89 nm. They applied nanocapsules on cotton fabric with 20% and 40% on the weight of binder using
90 pad-dry-cure method and the latent heat was found 15.2 Jg^{-1} and 19.1 Jg^{-1} g respectively.

91 The humans are thermally comfortable when their skin temperature is around 33 °C while in rest
92 and less than 33 °C if any of the physical activity is involved. No research has been focused until
93 now on synthesizing melamine formaldehyde NPCM in the range of 33 °C to target the skin
94 comfort temperature. The synthesis of nanocapsules in this research work focuses on the
95 preparation of nanocapsules using melamine formaldehyde as shell material and mixture of n-
96 octadecane and eicosane as core PCM. Moreover the developed capsules were in nano scale with
97 narrow size distribution suitable for melt spinning process for synthetic filament production to
98 overcome mixing and extrusion problems.

99 **Materials & Methodology**

100 Melamine and formaldehyde were purchased from Alfa Aesar® and used as raw materials to
101 synthesize MF (melamine formaldehyde) capsule shell. Formaldehyde purchased was 37% w/w
102 in aqueous solution. Eicosane and n-octadecane purchased from Alfa Aesar® were used as PCM
103 core material. Sodium dodecyl-benzene-sulfonate (SDS) was used as an emulsifying agent and
104 polyvinyl alcohol (PVA) was used as a stabilizer. Acetic acid and NaOH were used as pH
105 controllers. The synthesized nanoencapsulated paraffin was compared with commercially
106 available MPCM 28 which is pure octadecane containing phase change temperature of 28 °C.

107 The nanocapsules were synthesized and incorporated in polypropylene yarn through melt spinning
108 process and were applied on cotton fabric via pad-dry-cure technique. The experiments were
109 designed using Minitab and the effect of factors on response has also been studied.

110 DOE (design of experiments) aimed to identify the processing conditions, parameters and product
111 components that affect the quality of nanoencapsulation and optimize those factors for the best
112 result. DOE helps to investigate the effects of factors (input variables) on response (output

113 variables) at the same time. The designed experiments were composed of series trials where
114 different levels of input variables were used. Data was collected for each trial in the form of output
115 variables.

116 In the design of experiment, Box-Behnken method was selected for nanoencapsulation to
117 determine the effect of levels of different factors on response. Box-Behnken design usually has
118 fewer design points than central composite design thus it is less expensive to run with the same
119 number of factors. Box-Behnken design is very useful especially when safe operating zone of a
120 process is known. Box-Behnken design always has 3 levels per factor unlike CCD (central
121 composite design) which has up to 5 levels per factor because of the axial points in the design. The
122 axial points were not desired in the current research therefore Box-Behnken design was adopted.

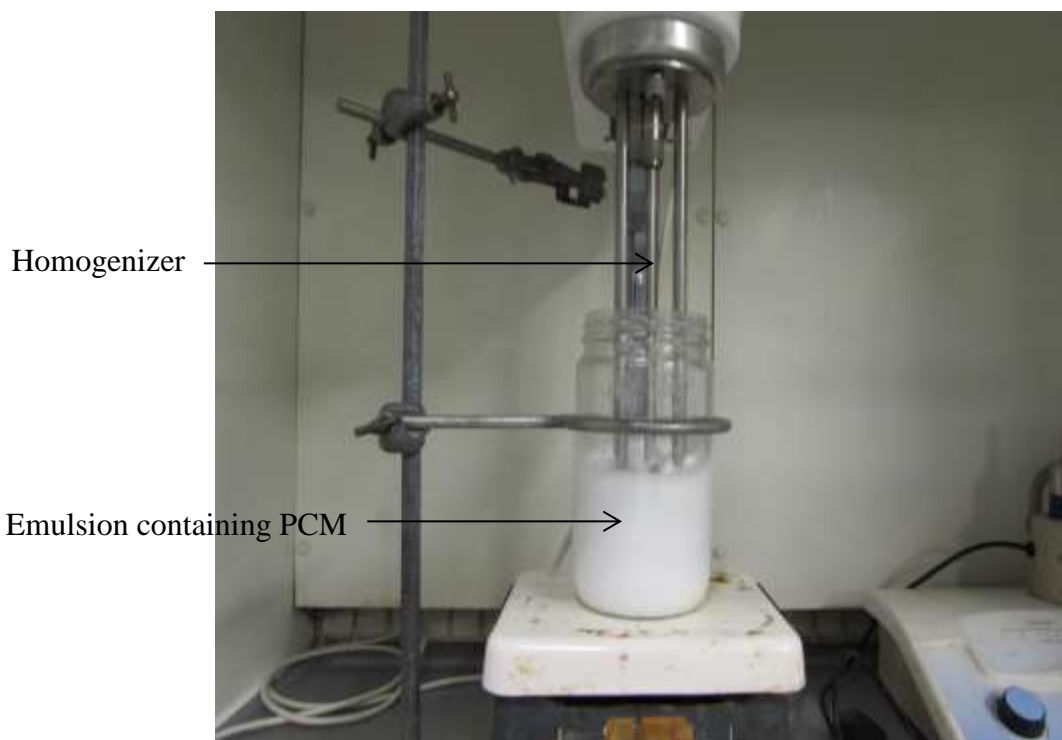
123 Table 1 shows the three factors and their levels which were used to study the particle size of
124 NPCM. Using the above lower and upper levels of each factor, experiments were designed using
125 Box-Behnken response optimizer. The estimated range of upper and lower levels was selected
126 based on the knowledge and experience to attain the desired response. Table 2 shows 15 designed
127 experiments in different combination of levels with each factor. All the experiments were
128 randomized by default and were performed according to the run order. The last column shows the
129 particle size against each experiment as outcome.

130 Table 1. Factors and levels for DOE

Factors	Lower level	Upper level
Emulsifier [g]	0.7	1.2
Emulsion speed [rpm]	5000	10000

134 **Experimental**

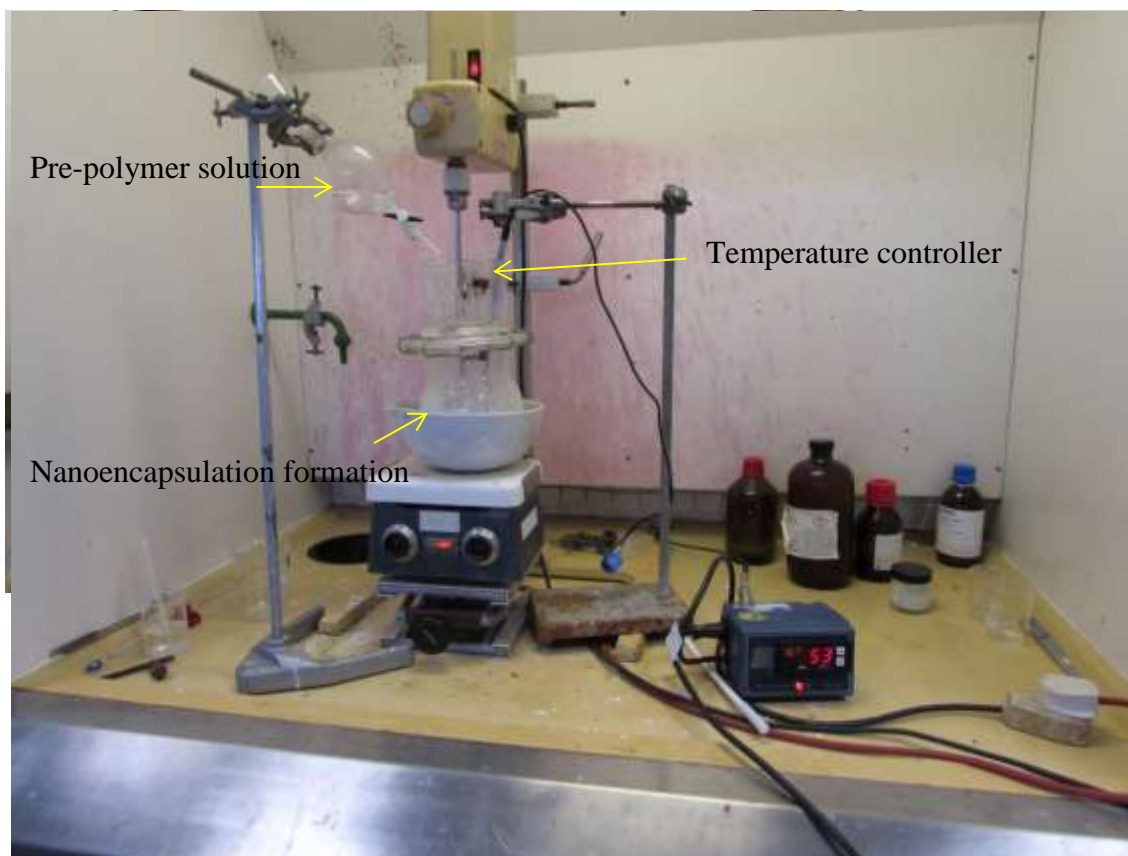
135 *In-situ* polymerization technique was used for the synthesis of nanocapsules. For oil in water
136 emulsion, (20g of) octadecane and (10g of) eicosane were taken in (150mL of) distilled water
137 containing SDS and (0.1g) stabilizer. The amount of SDS (emulsifier) and emulsion stirring rate
138 were used in different combination and has been shown in Table 1. The emulsion was prepared
139 under high stirring rate using high speed homogenizer at 70 °C for 30 to 40 min. as shown in Figure
140 1.



141

142 **Fig. 1** O/W emulsion using high speed homogeniser

143 The prepolymer solution was prepared by adding (10g of) melamine and (20g of) formaldehyde
144 in (50g of) distilled water and mixed at 60-65 °C as shown in Figure 2 (a) until a clear solution
145 was obtained as shown in Figure 2 (b).



146

147

Fig. 2 Pre-polymer formation using melamine and formaldehyde

148 For nanoencapsulation, the emulsion was poured into three neck flask and prepolymer solution

149 was dribbled into the emulsion drop wise as shown in Figure 3. The temperature of the mixture

150 was increased gradually up to 80 °C while controlling pH in the range of 4-4.5 using acetic acid.

151 The mixture was under stirring using different rpm speed as shown in experimental design. The

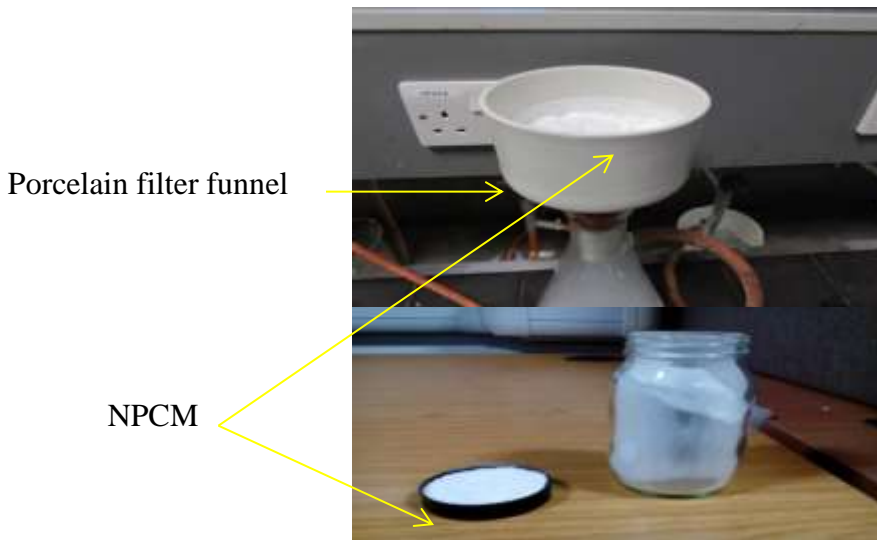
152 process was held under controlled parameters allowing polycondensation reaction to complete and

153 nanocapsules were washed, filtered and dried to get NPCM paraffin powder as shown in Figure 4.

154

155

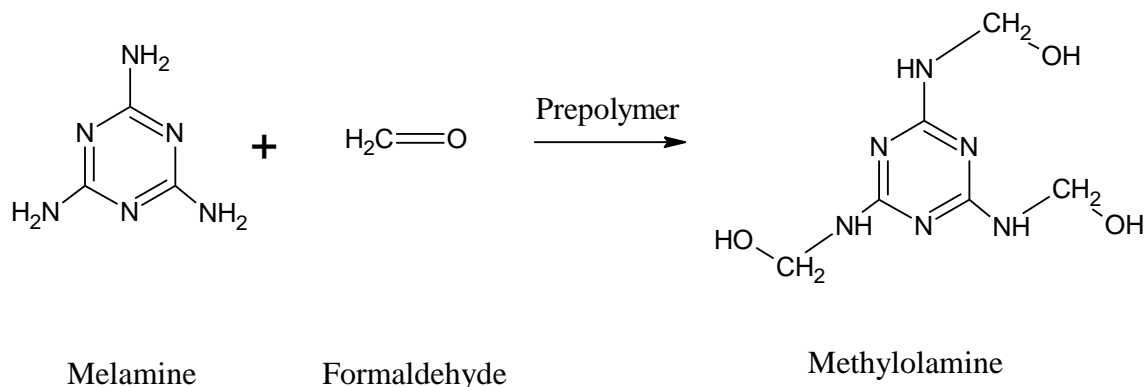
Fig. 3 Encapsulation of NPCM paraffin in reaction flask



157 **Fig. 4** NPCM after filtration and drying

158 Reaction mechanism

159 The first step is the direct reaction between melamine and formaldehyde forming methylolamine
 160 prepolymer as shown in Figure 5.

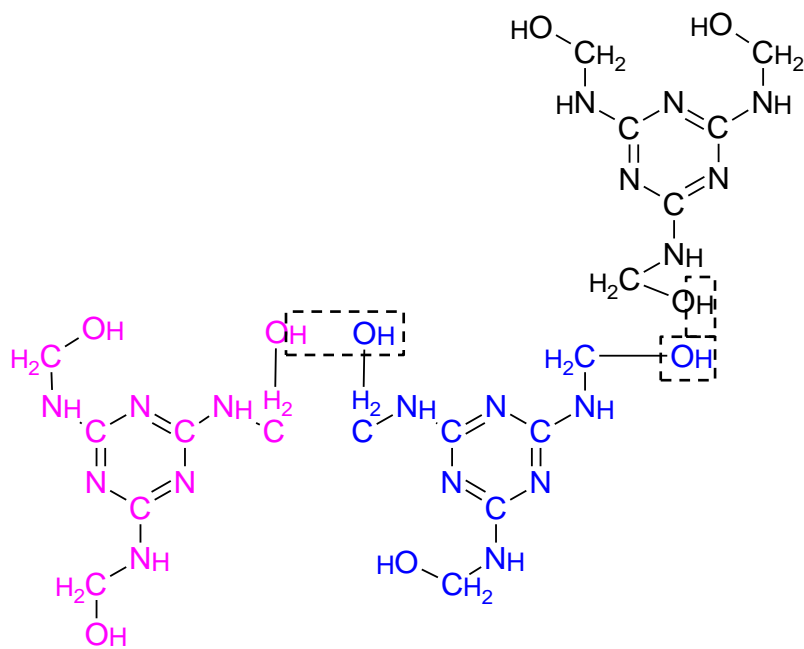


163 **Fig. 5** Formation of methylolamine

164 The second step shown in Figure 6 is called the condensation reaction step in which methylolamine
 165 combines together to form three dimensional network of cross linked melamine formaldehyde with
 166 methylene bridges. The condensation reaction takes place between the methylol groups of

167 neighboring methylamine with the elimination of water molecules which cross link in three
168 dimensions. The water molecules are shown in Figure 6 by dashed boxes which are eliminated
169 during the polymerization reaction.

170



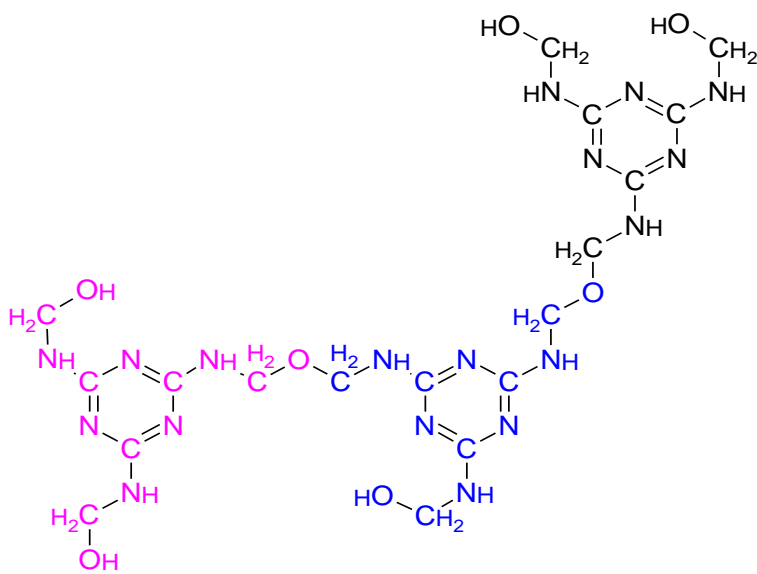
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174

-H₂O Condensation



175

176

Fig. 6 Condensation reaction of melamine formaldehyde

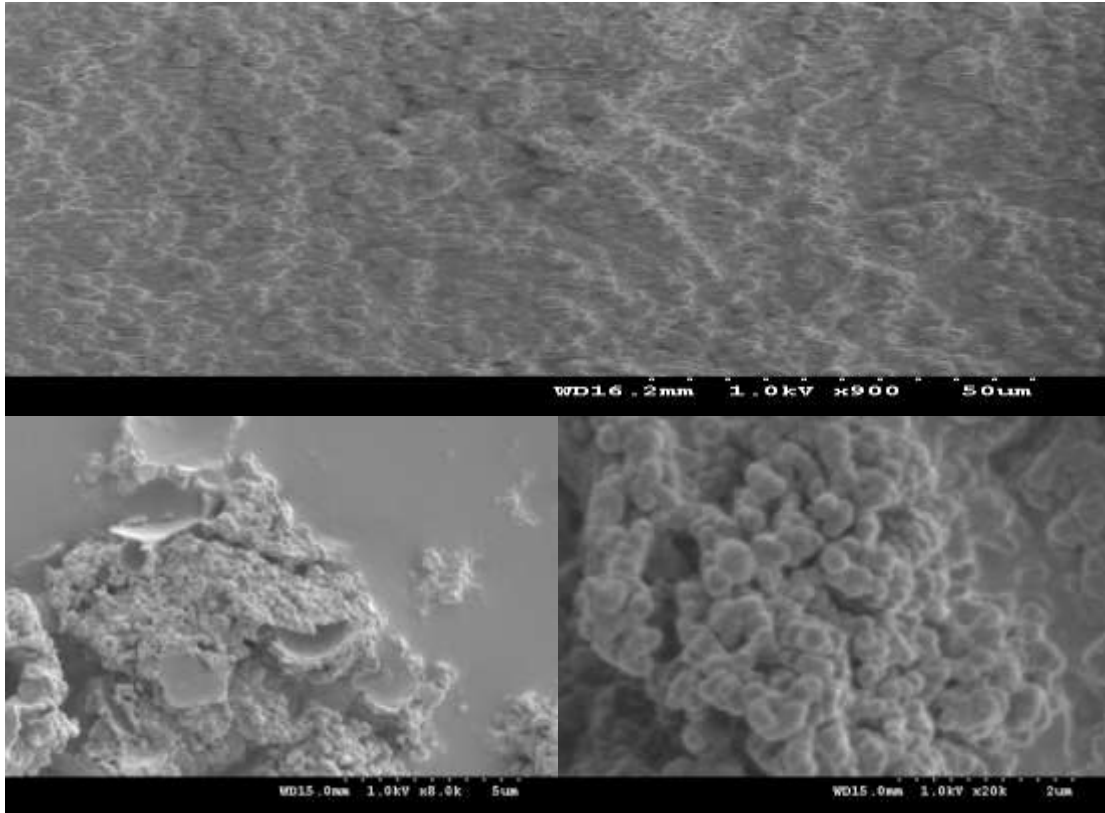
177 Application of NPCM paraffin on cotton fabric via pad-dry-cure technique
178 MPCM 28 and NPCM paraffin were applied on plain woven fabric made of 100% cotton by pad-
179 dry-cure technique. The coating method was not adopted because it makes the fabric stiffer and
180 adversely affects the breathability of fabric which is not ideal for the active smart textile market.
181 The padding solution contained: (60 gL⁻¹ of) polyurethane binder ARRISTAN EPD; (50 gL⁻¹ of)
182 REAKNITT ZF which is dimethyl-dihydroxyl-ethylene urea, formaldehyde free cross linking
183 agent which enhances the cross linking of binder; (50 gL⁻¹ of) TUBINGAL RGH softener which
184 is modified polysiloxane and (8 gL⁻¹ of) CHT CATALYST AD, a modified inorganic salt; and
185 MPCM 28 and NPCM paraffin (were used 30% of the weight of binder). The fabric was padded
186 with the pressure of 2 bars between the mangles with speed of 2 mmin⁻¹ using two dip two nips.
187 The fabric was dried (at 100 °C) and then cured in a stenter (at 150 °C for 3 minutes).
188 Each sample was then taken for washing (1 wash and 5 washes) to study the durability of the
189 applied PCM capsules. British Standard BS EN 26330: 1994 program 15, 6A 40 °C was followed
190 to perform the washing of samples. SEM and DSC were used to characterize the washed fabrics
191 as comparison to the untreated.

192 **Results and discussion**

193 SEM for nanocapsules

194 The images of nanocapsules were analyzed using scanning electron microscopy. The images of
195 NPCM paraffin are shown in Figure 7 which indicates that all the images are very consistent in
196 their particle size. The images shown in Figure 7 were taken at lower magnifications to show the
197 aggregates of nanocapsules with narrow size distribution. The images taken at 8000 and 20000

198 magnification in Figure 7 indicates that nanocapsules are very consistent in size and are spherical
199 in shape.

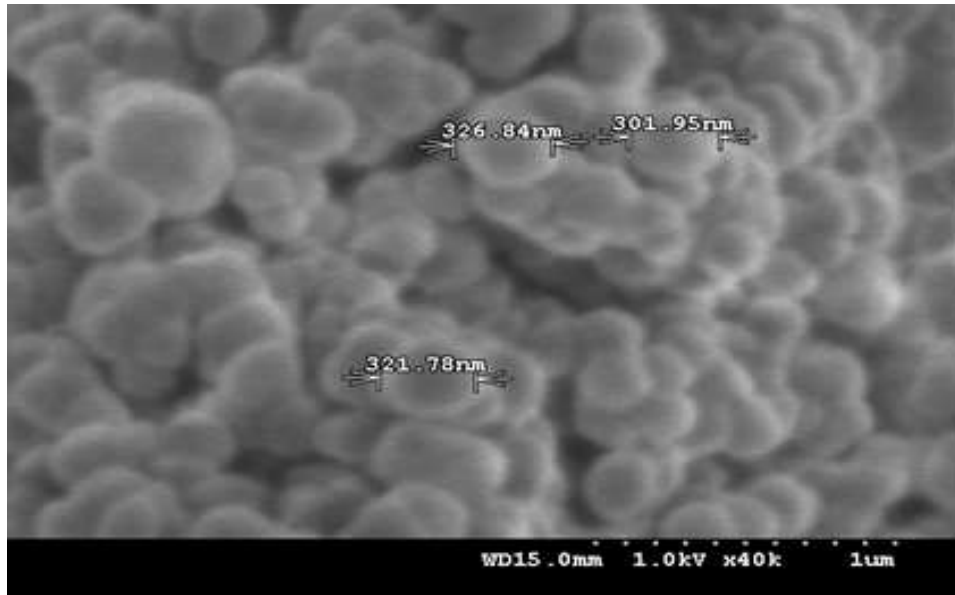


200

201

Fig. 7 SEM images of NPCM paraffin

202 Figure 8 shows the SEM images at higher magnification of 40K and were used to measure the size
203 of nanocapsules. The size of nanocapsules of one sample measured through SEM was from 300
204 nm to 326 nm as shown in Figure 8.



205

206

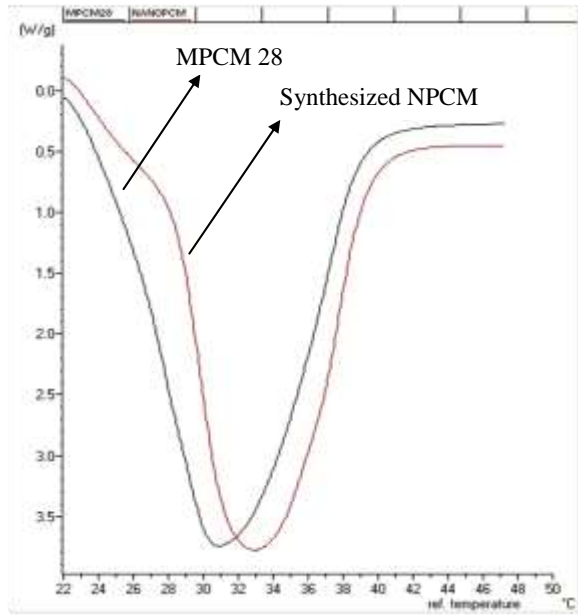
Fig. 8 SEM images at higher magnification

207 DSC study of NPCM paraffin

208 The result of latent heat from DSC shows the presence of paraffin in NPCM. The melting
209 temperature of NPCM paraffin was found near the human skin comfort temperature i.e. 33 °C.

210 Figure 9 shows the curves of MPCM 28 (from supplier) and NPCM synthesized in this research.

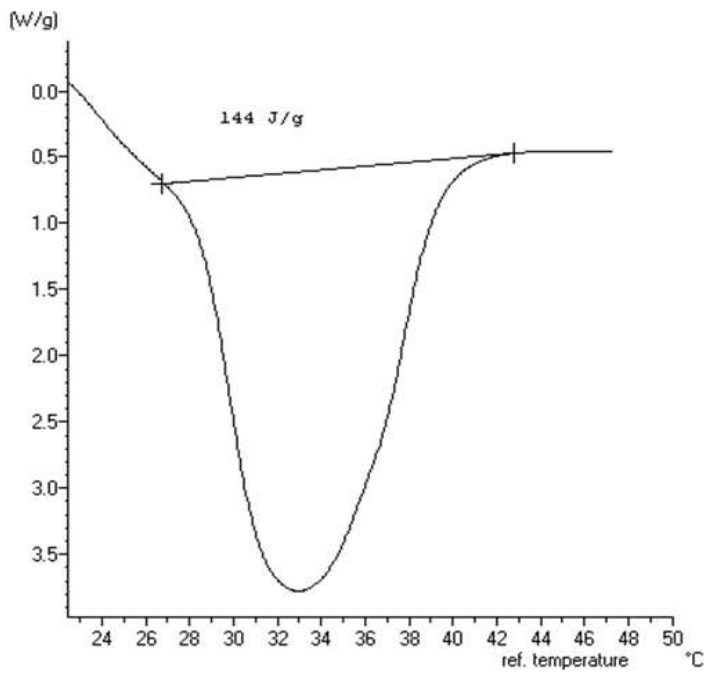
211 The peak melting temperature of both curves shows clearly that the phase change temperature of
212 the newly developed NPCM is closer to human skin comfort temperature although the curved areas
213 are almost the same. The latent heat of NPCM paraffin is 144 Jg^{-1} as shown in Figure 10.



214

215

Fig. 9 DSC curves for MPCM 28 and NPCM paraffin



216

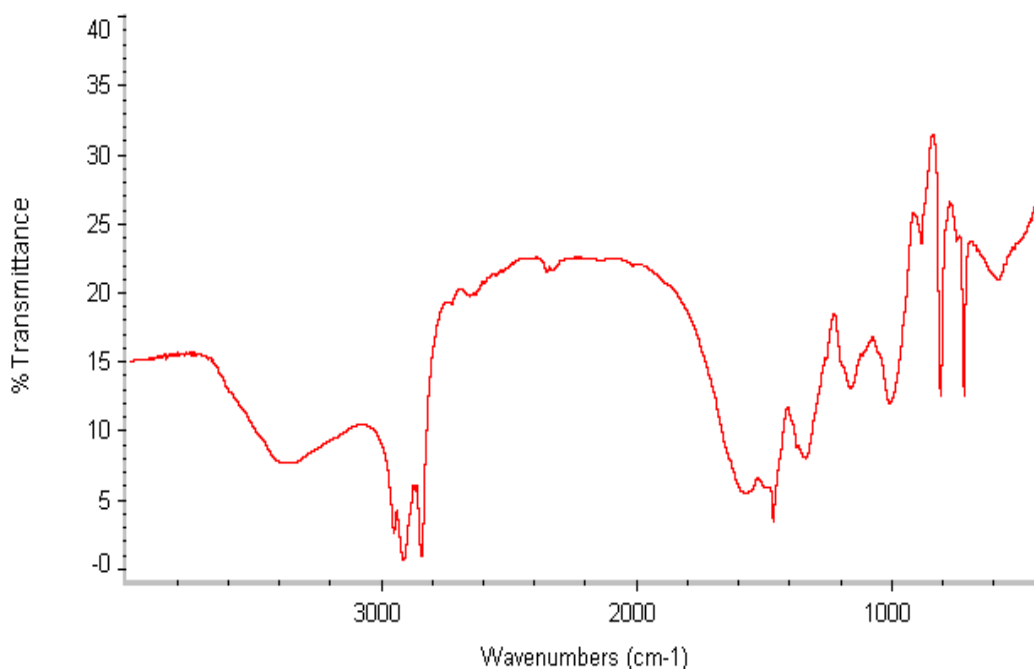
217

Fig. 10 Latent heat for NPCM paraffin

218

219 Structure of NPCM paraffin

220 The FTIR spectra of NPCM paraffin is presented in Figure 11. The spectrum shows a strong
221 absorption band at 2850-2930 cm^{-1} attributing to the stretching vibration of aliphatic C-H group of
222 the paraffin. The N-H stretching can be observed at 3300 to 3400 cm^{-1} and N-H bending can be
223 seen at 1320 cm^{-1} . The C-N stretching in the triazine ring can be seen at 1550 cm^{-1} and triazine
224 ring bending is presented by a very sharp peak at 810 cm^{-1} . The peak at 1150 shows the presence
225 of ether group which is the proof the melamine formaldehyde reaction. This proves that the
226 developed nanocapsules indeed are composed of paraffin as core and melamine formaldehyde as
227 shell materials.



228

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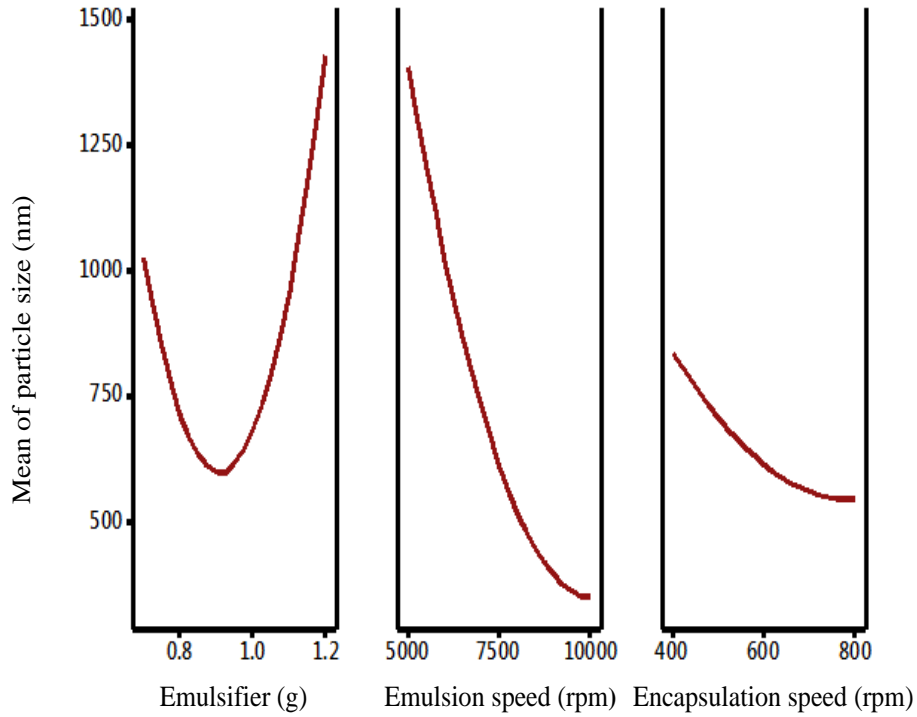
Fig. 11 FTIR for NPCM paraffin

230 Effect of factors on response of particle size

231 *Main effect plots for particle size*

232 Three parameters called the amount of emulsifier, emulsion stirring speed and encapsulation
233 stirring speed were studied to investigate the effect of nanocapsules particle size. The main effect
234 plot for particle size is shown against each parameter individually in Figure 12. The graph shows
235 that particle size decreases as the amount of emulsifier increases up to a certain level, then
236 increases when further increasing the amount of emulsifier. This is caused by emulsifier which has
237 functionality of enhancing the emulsification process by linking its hydrophobic tail to the oily
238 paraffin. As the amount of emulsifier increases, the emulsification increases hence the droplet size
239 decreases. After the saturation takes place, any addition amount of emulsifier starts agglomeration
240 among the individual mono-molecular droplets which affect the efficiency of emulsification
241 process by increasing the droplet size in the form of agglomerate. Hence the amount of emulsifier
242 is important to ensure the formation of nano-emulsion.

243 The second factor in the graph is emulsion stirring speed which plays the main role in the particle
244 size. The graph clearly indicates that increase in rpm decreases the droplet size which helps in
245 getting nano size capsules. The reason can be described as the stirring creates physical agitation
246 which keeps the emulsion in mono molecular form and even become more severe when the stirring
247 rate is increased.

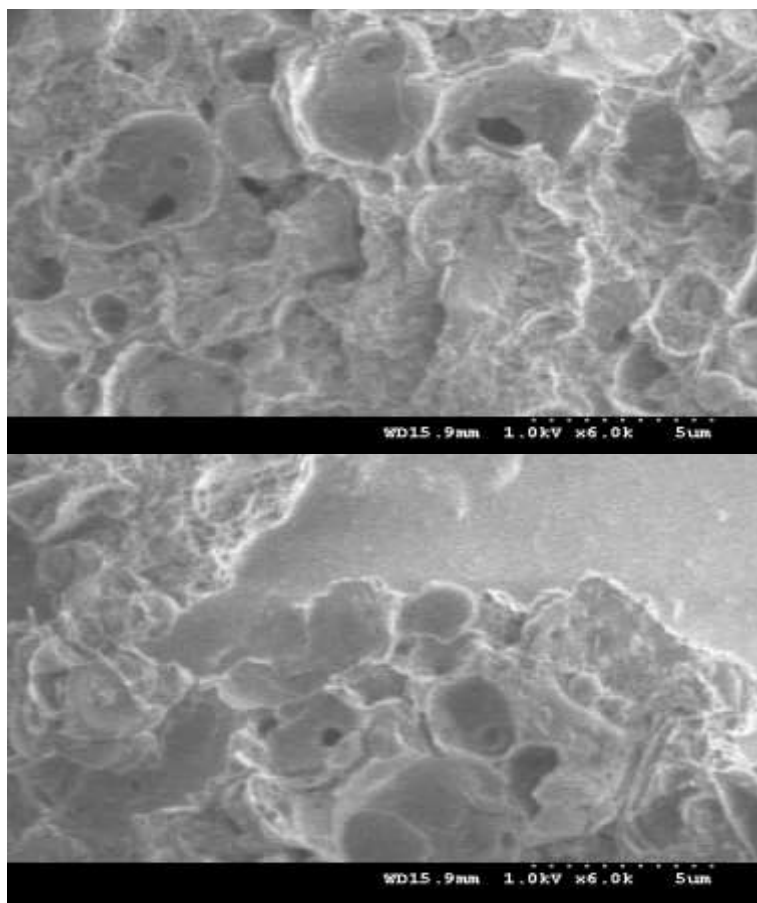


248

249

Fig. 12 Main effect plots for particle size

250 The third parameter was stirring speed during encapsulation process. Figure 14 shows that the
 251 stirring speed during nanoencapsulation also affects the particle size of NPCM. The effect of
 252 encapsulation stirring speed on particle size is less drastic than emulsion stirring speed. The
 253 optimum stirring speed is found to be 600 rpm for the encapsulation. As the encapsulation speed
 254 increased up to 800 rpm, the nanocapsules were found ruptured because of severe agitation and
 255 shear force of the stirring blades acting on capsules as shown in Figure 13.



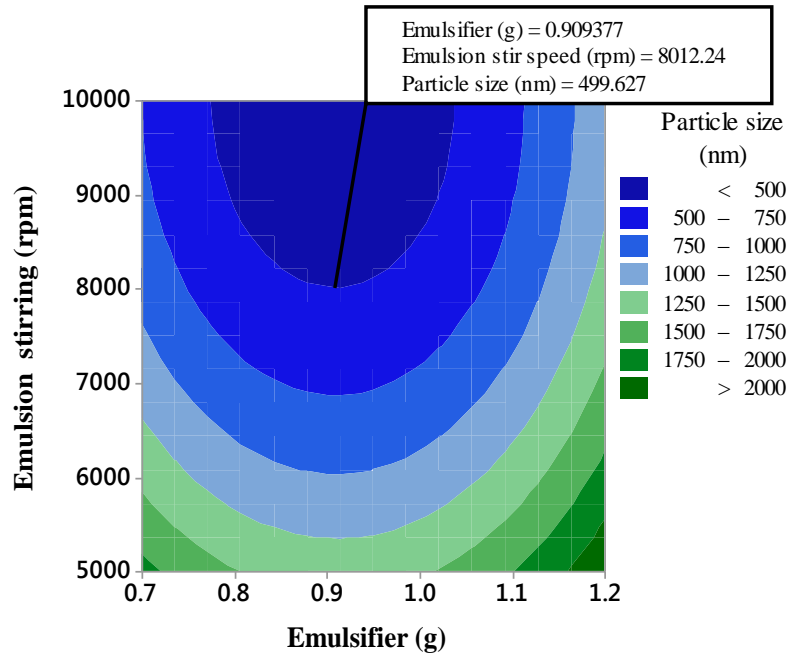
256

257

Fig. 13 Ruptured capsules with higher encapsulation speed

258 *Contour plots for particle size*

259 The contour plot shows how a response factor relates to the two continuous variables during
260 experimentation. With the help of contour plots, the relationship between outcome and variables
261 (parameters) can be determined. The contour plot in Figure 13 shows how emulsion stirring speed
262 and the amount of emulsifier influence on the size of the nanocapsules. The contour plot also helps
263 in determining desirable response values against the value of each factor. The particle size of 500
264 nm of NPCM paraffin can be achieved by using 0.9g emulsifier and around 8000 rpm emulsion
265 speed as shown in Figure 14. The above parameter values were taken by setting the value of third
266 factor (encapsulation speed) at 600 rpm.



267

268

Fig. 14 contour plot of particle size with emulsifier vs emulsion stir speed

269

Figure 15 shows the effect of emulsion speed and encapsulation speed on the particle size of

270

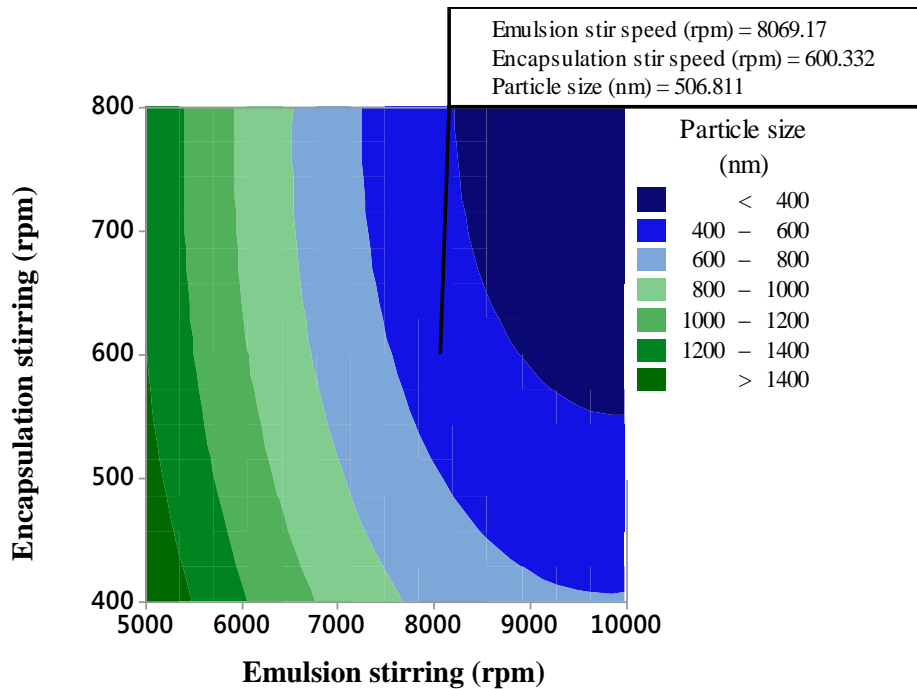
NPCM paraffin. The flag in Figure 15 shows the desired outcome of nanocapsules by setting the

271

emulsifier value at optimum level. If the size of particle is required for instance 500nm, then the

272

emulsion and encapsulation speed would be required around 8000rpm and 600 rpm respectively.



273

274

Fig. 15 Contour plot of particle size with emulsion vs encapsulation stir speed

275

Incorporation of NPCM paraffin in filament

276

The monofilament polypropylene yarn containing 4% NPCM paraffin was developed to study the

277

sustainability of nanocapsules in the filament. The developed filament was analyzed using SEM

278

and DSC and was compared to the previously developed polypropylene monofilament

279

incorporated with 4% MPCM 28.

280

SEM of NPCM paraffin incorporated filament

281

The SEM images are shown in Figure 16 which ensures the incorporation of NPCM within the

282

filament. The filament was cut diagonally cross-section wise in order to have better chance of

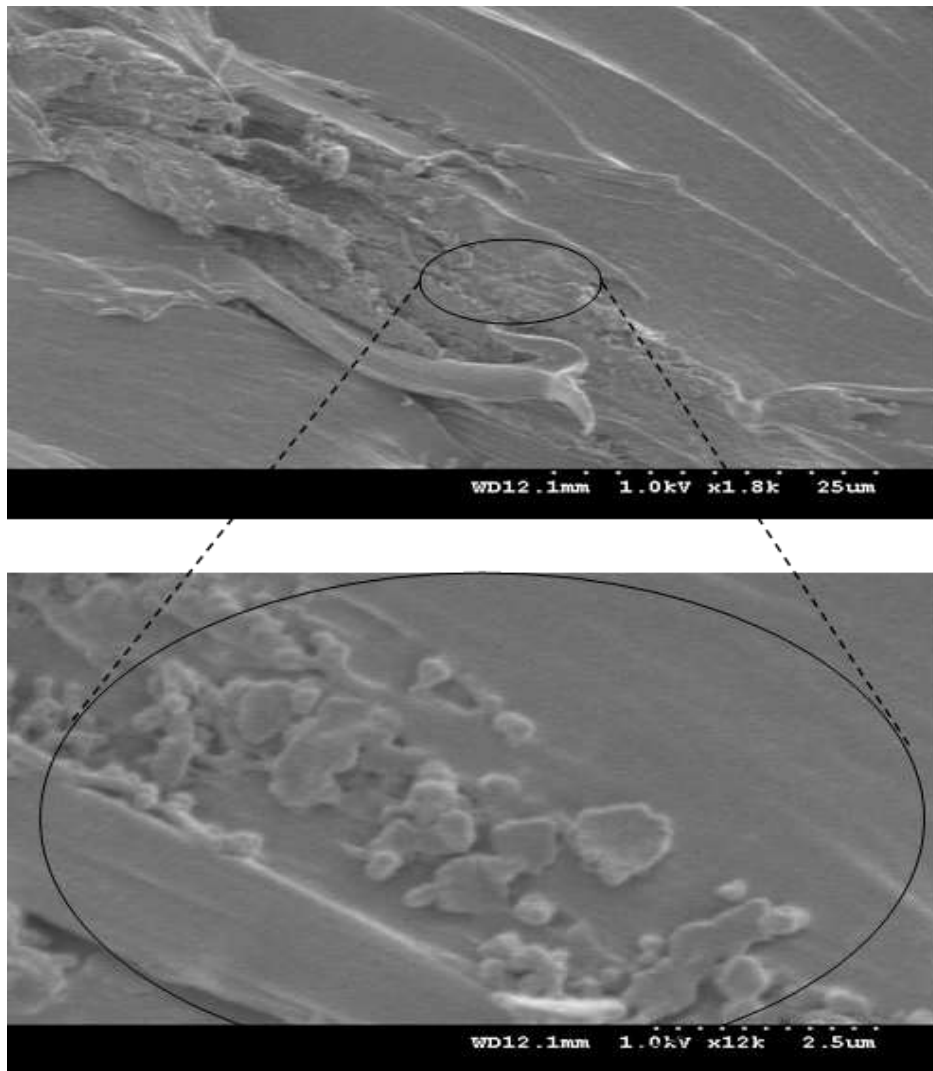
283

capturing nano capsules and images of nanocapsules were captured at different magnifications. It

284

is very clear from the images that the filament surface at its cross section is quite rough because

285 of the presence of nanocapsules within the filament. The image taken at higher magnification
286 shows the presence of nanocapsules scattered randomly.



287

288 **Fig. 16** Nanocapsules within the yarn at higher magnification

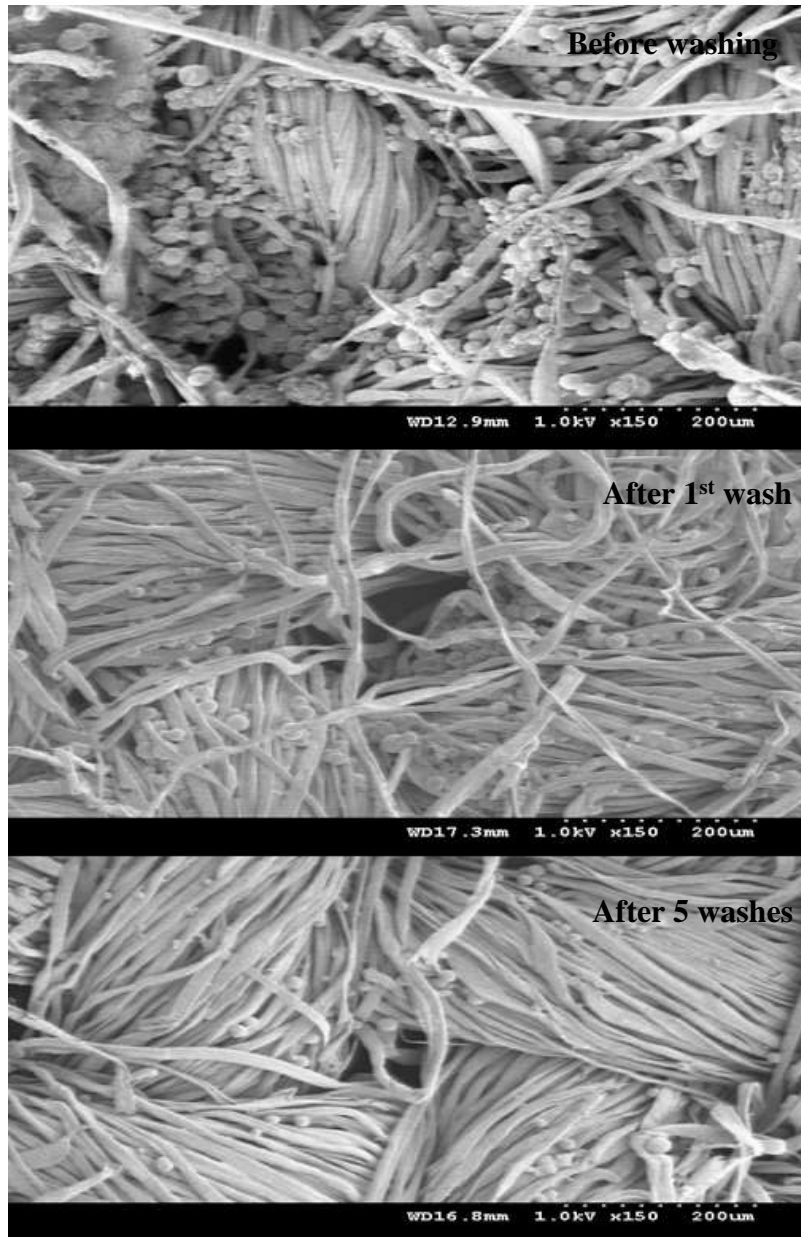
289 *DSC study of NPCM paraffin incorporated filament*

290 Latent heat for the filaments incorporated with 4% MPCM 28 and NPCM paraffin were tested
291 using DSC and were found to be 2.31 Jg^{-1} and 3.24 Jg^{-1} for filaments with MPCM 28 and NPCM
292 paraffin respectively. The reason for more latent heat in yarn incorporated with NPCM paraffin

293 can be described as the NPCM paraffin capsules are smaller as compared to that of the MPCM 28
294 which increases the incorporation of nanocapsules resulting in enhanced thermal characteristics.
295 The larger the size of capsules, the more difficult would be for capsules to be incorporated into the
296 filaments during extrusion process. Secondly during extrusion process surface deposition of
297 microcapsules occurred resulting in the capsules adhered to the surface of the filaments to be
298 removed easily in the subsequence drawing process. On the other hand, the nanocapsules are prone
299 to incorporate inside the yarn and even because of the smaller size they make integral part of the
300 surface which is not removed significantly during the subsequent process of drawing.

301 SEM for NPCM paraffin treated cotton fabric

302 Figure 17 shows the images of fabric finished with MPCM 28 before and after washing. The
303 microcapsules are spread all over the fabric after pad application and attached to the surface with
304 the help of binder. The images of the same fabric after 1st and 5 washes show that microcapsules
305 are still present but not in large amount especially for the fabric after 5 wash. When the capsules
306 are in large size, most of the capsules are attached on the surface of yarn and fabric but would not
307 sustain after severe washing. Only those capsules bound firmly to the yarns and fabric with the aid
308 of binder would not be washed off.



309

310

Fig. 17 Fabric treated with MPCM 28 before and after washing

311

Figure 18 shows the images of fabric treated with NPCM paraffin before washing. It is clear from

312

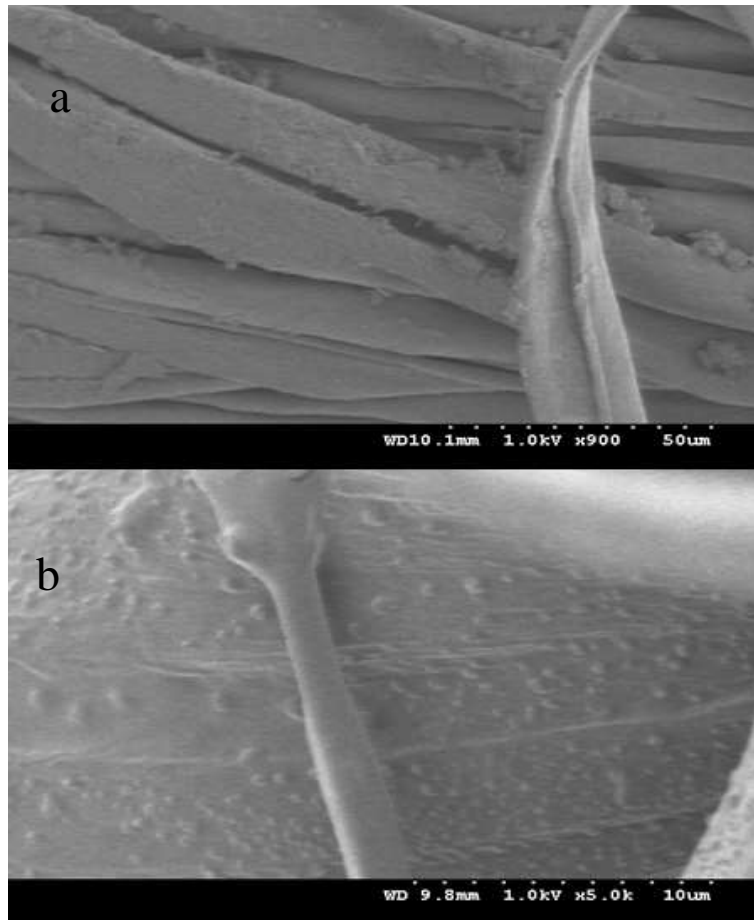
Figure 18a that large amount of nanocapsules are bound on the yarns of fabric. Figure 18b is a

313

magnified image which even more clarifies that nanocapsules are firmly bound to the yarn surface

314

and not just adhered to the fabric as was seen for the case of MPCM 28 treated fabric.



315

316

Fig. 18 Fabric treated with NPCM paraffin before washing

317

Figure 19 shows the SEM images of treated fabric after one wash. The nanocapsules are attached

318

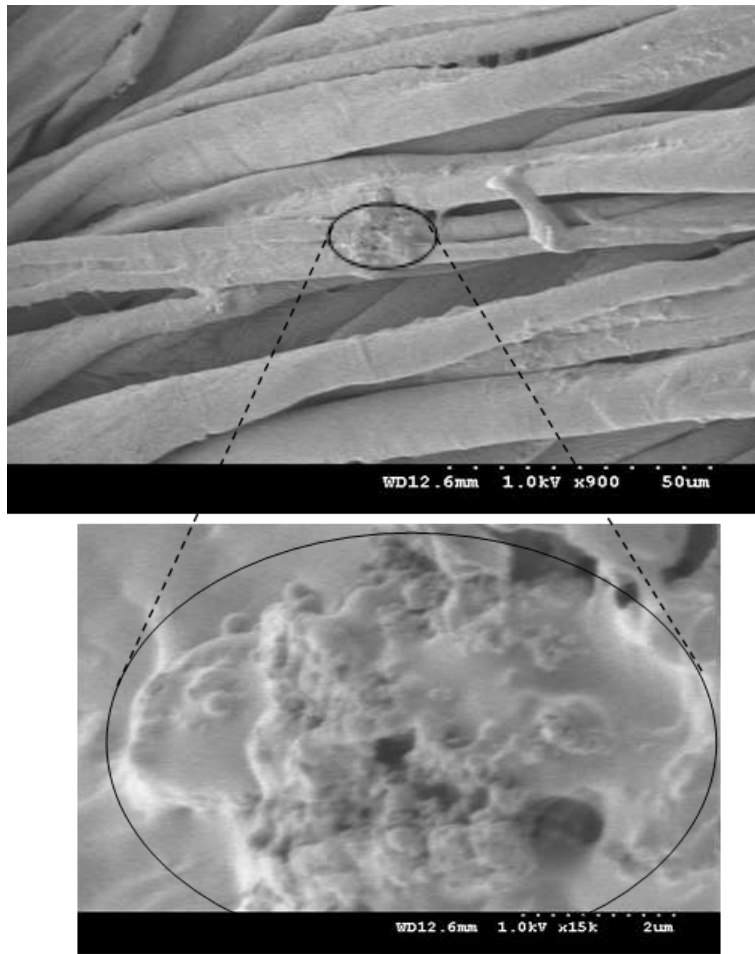
to fibre level in comparison to microcapsules which are attached to yarns of the fabric; they are

319

more firmly attached and perform better resistance to washing. The image in Figure 19 is

320

magnified to show how nanocapsules are mixed within the resinous material of binder.



321

322

Fig. 19 SEM for fabric treated with NPCM paraffin after 1st wash

323

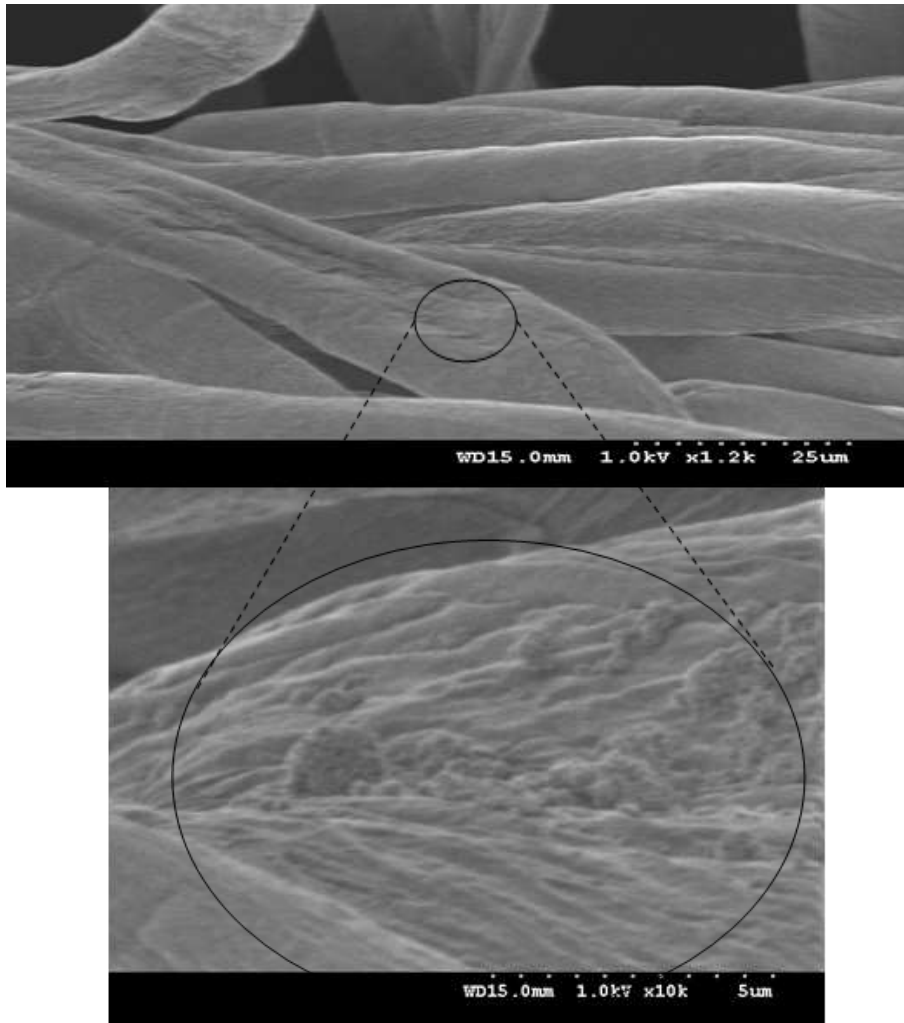
Figure 20 shows the images of fabric treated with NPCM paraffin after 5 washes. The nanocapsules

324

are still present in large amount on the surface of yarns up to the fibre level along with binder

325

because of the nano scale size.



326

327

Fig. 20 Fabric treated with NPCM paraffin after 5 washes

328

Figure 21 shows the comparison of images of fabric treated with both MPCM 28 and NPCM

329

paraffin in one bath. This is clear from the images that microcapsules with MPCM 28 are attached

330

to the surface of the filaments while the newly developed nanocapsules are bound to the fibre as

331

its integral part with the help of binder. As long as binder is present, the nanocapsules are difficult

332

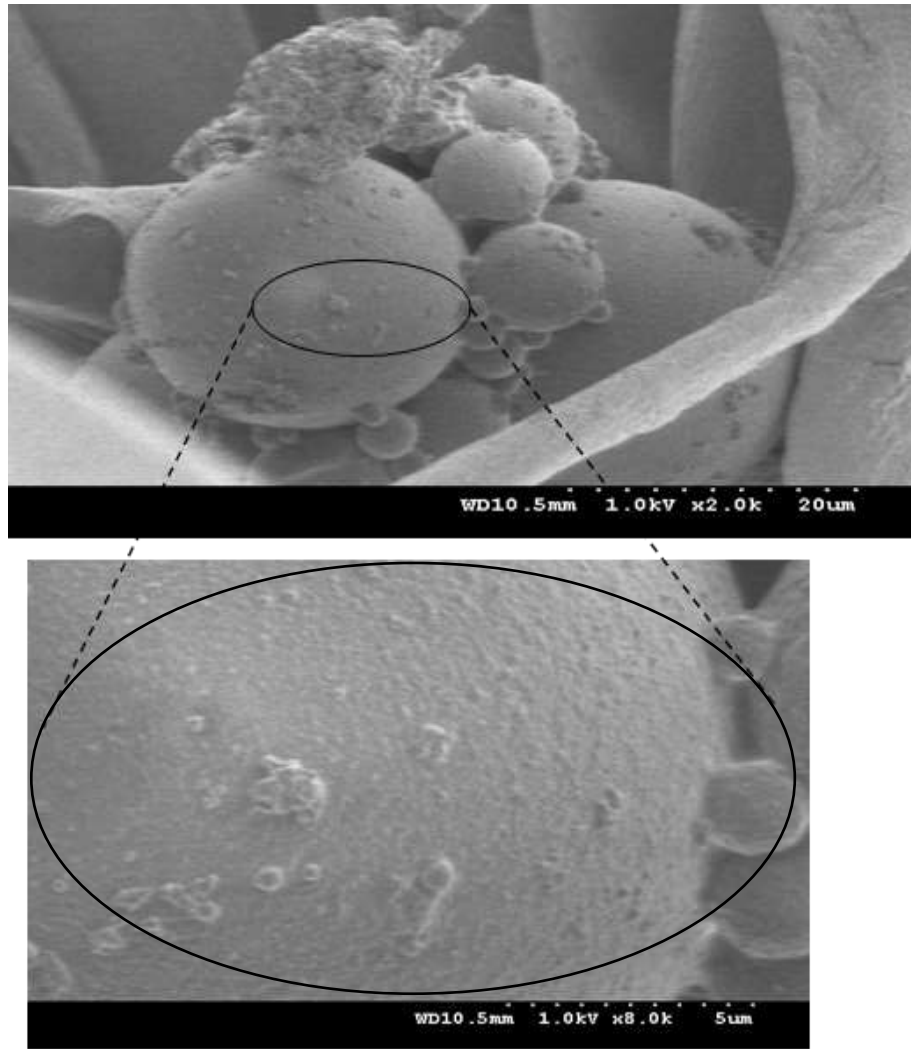
to be removed. In Figure 21 larger capsule is magnified showing that a significant amount of

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nanocapsules is spread even on the surface of microcapsule; this means if nanocapsules are used

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instead of microcapsules, they can enhance the thermal characteristic of the treated fabric.



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Fig. 21 Comparison of MPCM 28 and NPCM paraffin in one bath

337 *DSC study of NPCM paraffin treated fabric*

338 DSC was performed for all fabrics treated with MPCM 28 and NPCM paraffin before and after

339 being washed. Table 3 shows the values of MPCM latent heat in comparison to NPCM paraffin

340 latent heat after certain washes. Figure 22 shows the graph of DSC curves of treated cotton fabrics

341 with MPCM 28 and NPCM paraffin after one wash and after five washes in comparison to the

342 original. This is clear from Figure 22 that after washing the latent heat of the fabric decreases 60%

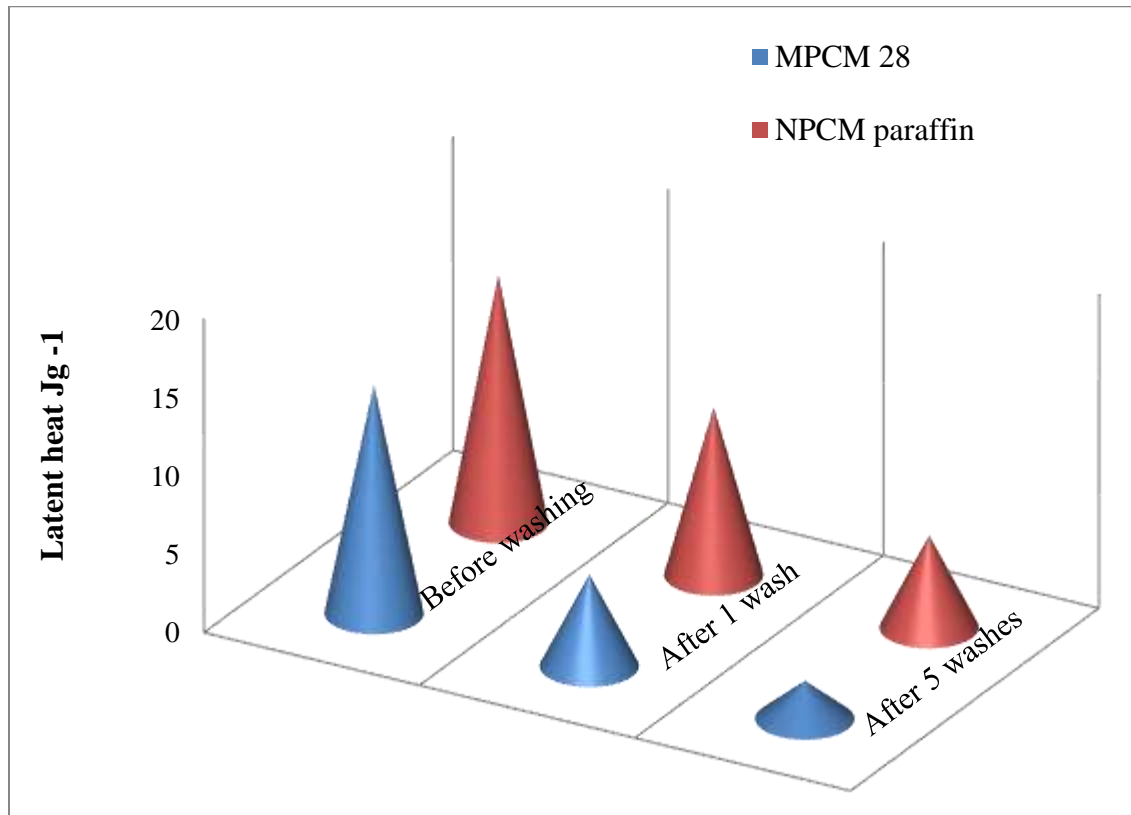
343 and 83.4% for the fabrics after one wash and five washes respectively in case of MPCM 28

344 (microcapsules) while the latent heat of cotton fabrics decreases 32.2% and 62.8% after one wash
345 and 5 washes respectively in case of NPCM paraffin (nanocapsules). The reason is that
346 nanocapsules are difficult to be removed as they bind well with filaments in the fabric due to the
347 nano scale of the particles. The smaller the size of capsules, the more firmly it bound to the
348 filaments in fabric. The developed nanocapsules are more durable to washing as compared to
349 commercially supplied microcapsules.

350 Table 3 Comparison of latent heat from DSC

Durability	MPCM 28 latent heat (J/g)	NPCM Paraffin latent heat (J/g)
Without washing	14.6	12.3
After 1 st wash	5.83	9.31
After 5 washes	2.42	4.64

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354 **Fig. 22** Comparison of latent heat of cotton fabrics treated with MPCM 28 and NPCM paraffin

355 **Conclusion**

356 The objective of this research was to synthesize and optimize the process of nanoencapsulated
 357 PCM and compare their durability with microencapsulated PCM after the application on cotton
 358 fabric. Also to synthesize capsules in the nano range using paraffin in combination and to bring
 359 the phase change temperature closer to the human skin. The NPCM paraffin were incorporated in
 360 to polypropylene fibre and was compared to the MPCM developed yarn. The NPCM paraffin was
 361 also applied on cotton fabric via pad-dry-cure method and following conclusions are drawn based
 362 on the analysis:

- 363 • The melting temperature of NPCM paraffin was found in the range of 32-33°C which is
364 closer to skin comfort temperature. The capsules formed were also in nano scale range and
365 the size obtained was in the range of 320 nm.
- 366 • The NPCM paraffin incorporated polypropylene showed 28% more latent heat than
367 commercial MPCM 28 incorporated polypropylene yarn.
- 368 • The application of nanocapsules on fabric showed better results than the microcapsules.
369 The fabric treated with NPCM paraffin showed 32.2% decrease in latent heat after one
370 washing while fabric treated with commercial MPCM showed 60% decrease in latent heat.
- 371 • The cotton fabric treated with nanocapsules shows better durability than microencapsulated
372 PCM due to its better adhesion with fibrous material of cotton. Hence the thermoregulating
373 cotton fabric with more durability has been developed using synthesized nanoencapsulated
374 paraffin.

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377 **Conflict of interest**

378 The authors of this research declare that they have no conflict of interest.

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