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Development of a Phase Change Material (PCM) Measurement Methodology for Fabric Surface Quantification

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ABSTRACT

Phase change materials (PCMs) are chemical compounds encapsulated in a polymer shell that have unique properties, allowing them to change phases from solid to liquid when a specific temperature is reached. As skin temperature rises, heat is absorbed and as the skin cools, heat is released. PCMs can be found in a wide range of consumer apparel and are often applied as a finish to the fabric. The durability of PCM finishes over the material or product's useful life is not well known, nor is there a standardized procedure for quantifying the presence of PCMs on the fabric's surface. The purpose of this research was to develop and analyze the efficacy of a microscopic evaluation methodology for fabric surface quantification of PCM finishes. A pilot study was conducted on 100% polyester athletic t-shirts with a proprietary printed PCM finish. Microscopic images were taken in three locations from sample shirts using a Nikon inspection microscope at new and after 1, 5, 10, 20, 25, 35, 40, 45, and 50 consumer launderings. Images were then analyzed using Adobe® Photoshop® tools. Two methods of identifying and quantifying PCMs within the images were explored, both ultimately finding the percentage of pixels containing PCMs within each image. Overall, the presence of the PCM on the fabric's surface diminished over the course of the garment's wash life. Results indicate that the methodologies developed within this study were effective for fabric surface quantification.

Keywords: Phase change material, thermoregulation, finish, durability, methodology

1. Introduction

Phase change materials (PCMs) are substances encapsulated in a polymer shell that have unique chemical compositions, allowing them to change phases from solids to liquids when a specific temperature is reached (*How Outlast Technology Works*, n.d.). This technology is a type of smart coating, which are fabric finishes that enable the material to sense and respond appropriately to environmental stimuli (Kanjana & Nalankilli, 2018). PCMs are either organic or inorganic and can be found as paraffins, fatty acids, salt hydrates, or metal particles, each with their own set of traits (Keyan et al., 2012). The process of changing from solid to liquid absorbs thermal energy, thus cooling the body, while the reverse change from a liquid to a solid releases thermal energy, heating the human body (Tyurin et al., 2018). Each type of PCM has its own unique melting point,

ranging from 8.1˚C to 130˚C (Keyan et al., 2012).

This type of active cooling technology was originally developed by NASA in the late 1980s for space suits and military applications and was later acquired by Outlast® and incorporated into a wide variety of end uses (NASA, 2009). PCMs have been modified and developed for multiple applications including heat storage systems for construction, home furnishings, and climate control for agricultural systems (Shin et al., 2005). Today, PCMs can also be found in a wide range of consumer apparel (McFarlin et al., 2016), including clothing for skiing, hiking, military applications, and activewear (Shin et al., 2005). In these applications, the purpose of PCMs is often to regulate the temperature of the body and to reduce any negative thermo-physiological effects of the environment (Keyan et al., 2012).

In textile applications, PCMs are often encapsulated in a permanent polymer shell (Tyurin et al., 2018). Microencapsulation is necessary in order to protect the PCM from the effects of its environment, prevent leakage, ease handling and application, and mask any potential odors or colors. These microcapsules are often between 20µm and 40µm in diameter (Keyan et al., 2012). While microencapsulation helps to prevent the breakdown of the PCM over its useful life, the longevity of the finish itself must be considered. Textile finishes may be classified by their useful life: temporary (last until the conclusion of the first cleaning cycle), durable (for the life of product but diminishes over time), or permanent (for the life of the product with no diminishment) (Kadolph, 2007). By using a binder to adhere the microencapsulated PCMs to fibers, the performance finish becomes durable, instead of temporary, allowing the consumer to benefit from the cooling effects during multiple wears. However, the PCM finishes are not permanent, meaning they do diminish over time. Therefore, there is a need in the industry to determine at what point and by how much the PCM is reduced after multiple types of wears and launderings. The efficacy of the PCM must be studied over the course of the product's wear life to ensure the remaining PCM is still providing benefits to the wearer, especially in cases where the finish is applied to personal protective equipment and is expected to improve the thermal comfort of the end user. In order to quantify the amount of PCM remaining on the fabric over the useful life of the garment, a simple and cost feasible measurement method is needed.

2. Review of literature

As thermoregulatory clothing continues to saturate the market (Tyurin et al., 2018), it is increasingly important that there be accessible ways to measure its performance. In order to effectively market fabric cooling technologies and provide a durable product for consumers, research must be conducted to ensure that PCMs are able to withstand and perform throughout the useful life of a garment. After an investigation of current literature, it can be noted that the durability of PCM finishes over the material or product's useful life is not well known. To the authors' best knowledge, there has been no documentation of a standardized procedure for quantifying the amount of PCM on a fabric's surface and monitoring its performance over time.

The ability to quantify the specific amount of PCMs present on the fabric's surface is important, as PCM cooling technology relies on next-to-skin surface contact in order to change the phase of the microcapsules on the fabric's surface. The following methods have been used previously to identify the presence of PCMs on the surface of fabric, however, each method has unique limitations in terms of the type of data that can be collected and instrument accessibility.

Scanning electron microscopy (SEM) has been cited in multiple studies as a way to obtain images of PCMs on the surface of the fabric (Lu et al., 2017; Shin et al., 2005). Shin, Yoo, & Son (2005) used SEM to photograph images of the

microcapsules on a fabric after being laundered in varying conditions (Shin et al., 2005). While SEM can detect the existence of the PCM and provide more insight regarding the microstructure, composition, and morphology of the PCM (Lu et al., 2017), this method is not capable of quantifying the specific amount of PCM present on the fabric's surface. Another disadvantage of this methodology is the cost of a scanning electron microscope which is approaching $1 million, with accessories (Brake, 2010). This is most likely out of the cost feasible range for the majority of textile testing laboratories, apparel suppliers, and clothing brands.

Lu, Sun, Chen, & Gao (2017) used optical transmittance spectroscopy to evaluate the colorfastness of the PCM coating over the course of repeated wash cycles. The comparison of results from this method ultimately aided the researchers in assessing the bonding strength of the PCM microcapsules (Lu et al., 2017). While this method aids in understanding how bond strength has changed over various washing intervals, it does not directly quantify the PCMs in terms of the amount of microcapsules on the surface.

Another method, gas chromatography mass spectrometry (GC/MS) requires the user to know the chemical composition of both the PCM, the binder, and the fibers within the cooling finish in order to separate and quantify the varying compounds (Bull, n.d.). While this method would provide an accurate way to specifically quantify the amount of PCMs on the surface of the fabric, it is often not feasible for apparel applications as these finishes can be trade secrets of fabric converters (finishers). The chemical knowledge needed for the GC/MS method is often not disclosed to the supply chain customer, preventing this type of evaluation in most applications. In addition, this method is also heavily limited by the high cost of instrumentation.

Fourier-transform infrared spectroscopy (FTIR) uses infrared spectroscopy to produce data on the spectral characteristics of a sample. Depending on the sample, certain wavelengths are absorbed while others pass through. FTIR converts and presents this data (Bradley, n.d.). This is often useful for identifying a chemical compound or understanding how much of a known compound is present in a sample. Similar to GC/MS, in order to use this method the user would need to know the specific chemical composition of the PCM and of the fabric in order to accurately distinguish and quantify differences between the two. As this information is rarely available to the supply chain customer, this method is also heavily limited.

Another method utilized by Shin, et al. (2005) was differential scanning calorimetry (DSC) which measures thermal properties (Shin et al., 2005). This method identifies differences in heat storage capacity between finishes with various concentrations of PCM. Shin, et al. were able to identify a decrease in the heat storage capacity when less PCMs and more of the binding material were present (Shin et al., 2005). While the previous study did not utilize this method to quantify PCMs between washes, DSC does have the ability to demonstrate the function of the PCM over multiple consumer uses and launderings. DSC is also more cost feasible than the instrumentations previously discussed, starting at $18,000 for a new calorimeter (*IKA C200 Calorimeter 230V 15-3/4"W x 15-3/4"H x 15-3/4"D (400 x 400 x 400 mm)* , n.d.). However, this method would not provide visual indications of the change in the presence of the PCMs on the fabric's surface, which are beneficial to ensure even spreading of the finish across the fabric and product's surface.

Finally, Bayés-García, et al., (2010) used thermo-optical microscopy (TOM) to obtain more accurate measurements of the physical geography of the PCMs. The diameter and observations regarding the shape of the microcapsules were recorded (Bayés-García et al., 2010). This method yields important information, as the size of the PCM microcapsules is often directly related to its specialization. Different types

of PCMs (paraffins, salt hydrates, etc.) each have different sets of traits such as melting points, duration of the heating or cooling effect, and economic feasibility. These traits can also be affected by the size and morphology of the PCM (Tyurin et al., 2018). Different textile applications may require different traits, therefore, being able to evaluate the physical characteristics of a PCM can aid in picking the appropriate PCM type, shape, and size for the application. However, this method only provides data on the PCM in its finish state, before it is applied to the surface of a fabric, and does not yield a measurement methodology for quantifying the amount of PCM that is present once applied to a textile product.

Despite the amount of options available to identify and characterize PCMs in various states, very few of these methods provide a means to quantify the specific amount of PCMs on the surface of a fabric in an economically feasible manner. Fabric surface quantification is a vital data point for multiple clothing applications in which PCMs act as a coolant to improve the thermo-physiological comfort of the wearer. Due to the impermanence of PCM finishes, it is important for designers, product developers, manufacturers, and end users to understand the longevity of the finish's performance. The devices used previously to characterize PCMs are often cost prohibitive and inaccessible to many labs and clothing brand companies, especially entrepreneurial start-ups. To the researchers' knowledge, a reliable, valid, and cost feasible method for quantifying the presence of PCMs on a fabric's surface has not yet been established. Such a methodology is needed to evaluate the durability of PCM finishes as a part of the quality control process. Therefore, the purpose of this research was to develop and analyze the efficacy of a PCM surface quantification methodology that is both accessible and reliable. Hence, a microscopic evaluation methodology was developed and assessed.

3. Methodology

3.1 Laundering durability study

In order to evaluate the longevity of a PCM finish over the course of a garment's wash life, a pilot durability study was conducted on 100% polyester athletic t-shirts with a proprietary printed encapsulated PCM finish applied to the back side of the fabric. A 4lb load consisting of six t-shirts with the PCM finish and six control t-shirts, without the PCM finish, underwent 50 consumer launderings. The specimens were loaded in a top-load, central agitator washer and dried in a residential electric dryer. The washer was set on a normal care cycle and filled with cold water and 35g of liquid consumer detergent. The load was dried on a timed cycle for 40 minutes on low heat. This process simulated the typical consumer usage of this product per the garment's care label instructions.

Evaluations of garment performance and PCM durability were performed prior to laundering and after 1, 5, 10, 20, 25, 35, 40, 45, and 50 wash cycles. After conditioning for a minimum of four hours, evaluations were performed on a minimum of three specimens per t-shirt type, including microscopic imagery. This manuscript focuses exclusively on the visual analyzation and quantification of the PCM finish, collected through microscopic imagery, which is outlined below.

3.2 Microscopic imagery of PCM finish

A printed PCM sample and a control sample were marked in three locations prior to testing: 1) the chest region, 2) near the bottom hem of the garment, and 3) on the sleeve. The rectangular locations were treated as a coordinate plane and each microscopic image was taken ten pixels to the right and ten pixels above the lower left corner of the rectangle. Images were taken in the same coordinate locations at each evaluation interval using a Nikon inspection microscope (iNEXIV VMA-2520 model) at 120X magnification (Fig. 1).

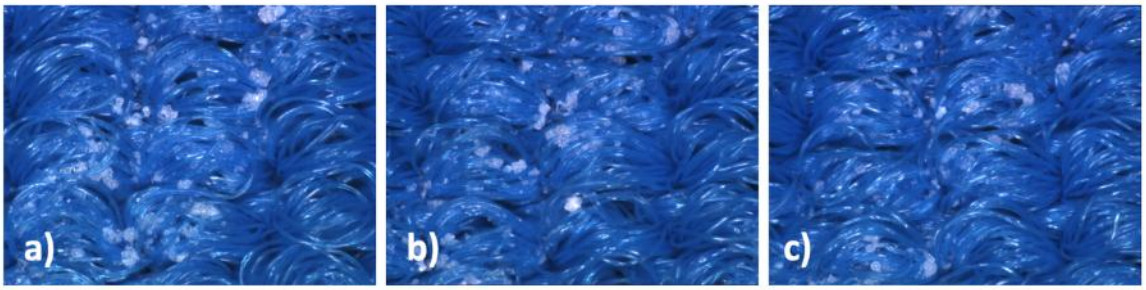


Figure 1. Images of the printed PCM finish on sample #1 at a) new, b) after 25 washes, and c) after 50 washes (from left to right).

3.3 Initial Photoshop® PCM analysis pilot trial

In order to visualize and digitally quantify the amount of PCM on the surface of the fabric, each microscopic image containing PCMs was converted into an Adobe® Photoshop® file. Prior to quantification, the exposure and sharpness of the image was increased in order to enhance visibility. The Photoshop® histogram window was then opened and the amount of pixels in the entire image was recorded. A new layer was created on top of the background and the brush tool was used to paint over all recognizable regions of PCM on the surface of the fabric with RGB red (Fig. 2).

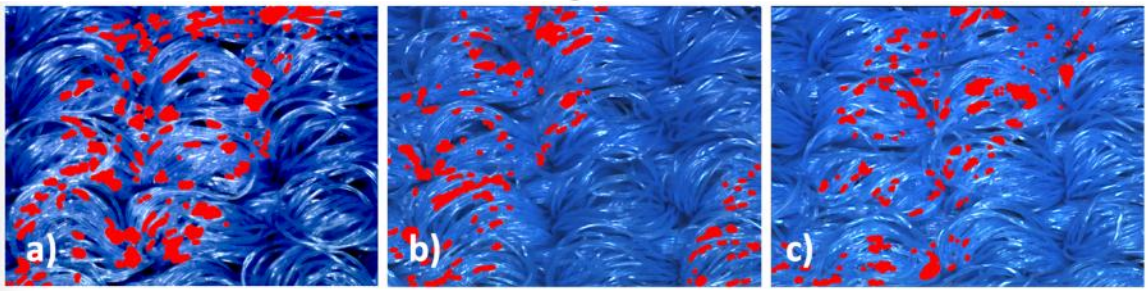


Figure 2. RGB red used to paint over encapsulated PCMs visible on the surface of the fabric at a) new, b) after 25 washes, and c) after 50 washes.

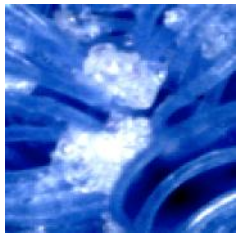
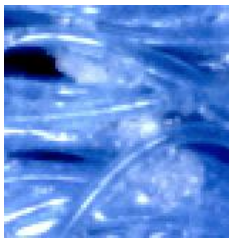
The histogram window was then opened a second time and the "channel" was set to "RGB Red" and the "source" was set to "selected layer". This displayed the amount of red pixels in Layer 1, which represented the amount of PCMs on the surface of the fabric. The amount of pixels were then recorded and the process repeated for all three PCM images at every testing interval. The average percentage of PCM pixels versus the number of total pixels in the initial image was calculated and recorded for each test interval. Results were analyzed and a need for further analysis with a more refined procedure was identified as, in some cases, the PCM clusters appeared as fuzzy or blurry spots between fibers, making it difficult to distinguish their definitive presence on the surface of the fabric.

3.4 Refined Photoshop® PCM analysis trial

During the pilot trial, several vague regions were identified and quantified as PCMs. In order to increase the precision and accuracy of the analysis, and avoid quantification of non-PCM regions on the surface of the fabric, specific standards for what constitutes the presence of a PCM were introduced. Table 1 provides examples of the criteria used for clearer quantification of PCMs on the fabric's surface when counting pixels that depict the encapsulated PCM. In comparison to the pilot trial, this approach prevents counting errors caused by vague imaging.

A limitation of imposing these strict standards is the potential to under quantify all PCMs present within the fabric (e.g. those that are not clearly visible on the fabric's surface). However, given that surface contact with the skin is necessary in order to activate the PCM and transfer heat from the body to the fabric and vice versa, the researchers deemed quantification of PCMs with complete visibility on the fabric's surface as the greatest priority during the development of this analysis method.

Table 1. Classification chart used to guide the identification and quantification of PCMs on a fabric's surface.

Classification	Sample Images/Description
Quantified as Fabric Surface PCM	  Lightness of the PCM is distinct and the clusters have clear borders, separating them from the surrounding fibers.
Unidentifiable/ Not Located on the Fabric's Surface	  No clear borders to distinguish where the encapsulated PCM ends and the fibers begin.

The same microscopic images from the first trial were re-analyzed using the classification standards in Table 1 for fabric surface presence of the PCMs. Prior to analyzing, the exposure and sharpness of the images were increased to the same degree that they were in the first trial and the images were then converted into Photoshop® files. The histogram window was opened and the total amount of pixels in the image was recorded. PCMs were identified as regions that contained clear spherical shapes, had a higher amount of white light reflection than surrounding areas, and had clear borders between the PCM microcapsules and the fibers of the fabric. The "quick selection tool" was used to automatically identify the borders of a shape. This tool enabled quick identification of distinct shapes versus those which were too vague to be confidently quantified as a PCM on the surface of the fabric. With few exceptions, only shapes that were identifiable with this tool were counted as PCMs.

Once all PCMs that met the criteria of the new standard were selected using the "quick selection tool", the "select and mask…" feature was used and the output of the tool was set to "new layer." A new layer containing only the selected PCMs was created. The histogram window was opened and the "source" set to "selected layer." The "quick selection tool" was then used again

to select all shapes in the layer which isolated all of the PCMs and allowed the histogram window to display the number of pixels selected. The number of pixels that clearly contained images of PCMs distinctly present on the fabric's surface was quantified. This number was recorded and the process repeated for all three PCM images at every laundering assessment interval. The average percentage of the PCM pixels versus the number of total pixels in the initial image layer was again calculated and recorded at each wash interval.

3.5 Statistical analysis

To determine the statistical significance between the results after multiple wash intervals, two-sample T-tests, assuming equal variance, were performed using the basic statistical software package available in Microsoft Excel. A p-value of 0.05 was chosen to indicate statistical significance. Individual paired T-tests were used to determine the significant difference in the amount of PCM present on the surface of the fabric between each wash interval, as well as, over the course of the wash life (compared to the amount present at new, prior to laundering).

4. Results

4.1 Initial Photoshop® PCM analysis pilot trial

The average percentage of PCMs on the surface of the fabric after each laundering interval was calculated from the percentage of PCMs in all three locations on the t-shirt. The average percentage of PCMs present on the fabric's surface after each laundering interval is presented in Table 2.

Table 2. Average percentage of PCMs present on the fabric's surface after each wash interval using the original pilot analysis method.

Wash Interval	Average % of PCM on Fabric Surface
New	11.11%
After 1 Wash	9.81%
After 5 Washes	8.14%
After 10 Washes	11.21%
After 20 Washes	12.18%
After 25 Washes	8.23%
After 35 Washes	6.61%
After 40 Washes	7.12%
After 45 Washes	9.13%
After 50 Washes	7.79%

After 50 consumer wash cycles, there was a 3.32% average decrease in the amount of PCMs detected on the fabric's surface using the initial pilot analysis method. There was an initial 1.3% decrease in PCM after the first wash, however, average percentages continued to fluctuate throughout laundering, between quantification intervals. Two sample T-tests were run, comparing each wash interval to the one immediately following it, to see if the differences in the average percentage of PCMs were statistically significant (p <0.05). None of the percentage differences between intervals were determined to be statistically significant. Additional T-tests comparing the change in PCM presence across the garment's wash life (to before laundering) indicated that the difference between the average percentage of PCM on the fabric's surface at new and after 50 washes was not statistically significant (p <0.05). The largest measured reduction in PCMs on the fabric's surface, utilizing the initial pilot quantification method, occurred after 35 washes (-4.5%), demonstrating a non-linear pattern. The non-linear nature of the results, as well as the subjectivity of the procedures used to quantify the fabric

surface presence of the PCMs, indicated a need to further improve the methodology.

4.2 Refined Photoshop® PCM analysis trial

The second PCM analysis trial was developed to ensure the results were accurate and reliable, and to improve the overall methodology. The average percentage of PCMs present on the fabric's surface after each laundering interval using the standard visual criteria and Photoshop® "quick selection tool" is presented in Table 3.

Table 3. Average percentage of PCMs present on the fabric's surface after each wash interval using the refined analysis method.

Wash Interval	Average % of PCM on Fabric Surface
New	3.53%
After 1 Wash	3.59%
After 5 Washes	3.44%
After 10 Washes	4.02%
After 20 Washes	4.21%
After 25 Washes	3.03%
After 35 Washes	2.89%
After 40 Washes	2.92%
After 45 Washes	3.13%
After 50 Washes	2.24%

After 50 consumer laundering cycles, the average percentage of PCM on the surface of the fabric decreased by 1.29%. Two sample T-tests comparing each interval to the one immediately following it determined none of the differences in fabric surface quantification between wash intervals were statistically significant, similar to the pilot trial analysis. Likewise, the difference between the percentage of PCM on the fabric's surface at new and after 50 consumer laundering cycles was also not found to be statistically significant.

The quantified PCM fabric surface averages fluctuated in a similar pattern for both analysis trials, as shown in Figure 3. However, the variability between these fluctuations was reduced with the refined trial by increasing the selectivity when identifying and quantifying the PCMs within the image. This reduction in variability is demonstrated by the reduced size of the error bars for the second trial, as illustrated in Figure 3. It is also possible, and likely, that the slight increase in the percentage of PCMs on the fabric's surface after 10-20 washes could be due to the surface migration of PCMs within the shirt, as well as, the deposition of PCMs from other t-shirts in the same laundering cycle. Further research and analysis should be conducted to answer these additional research questions.

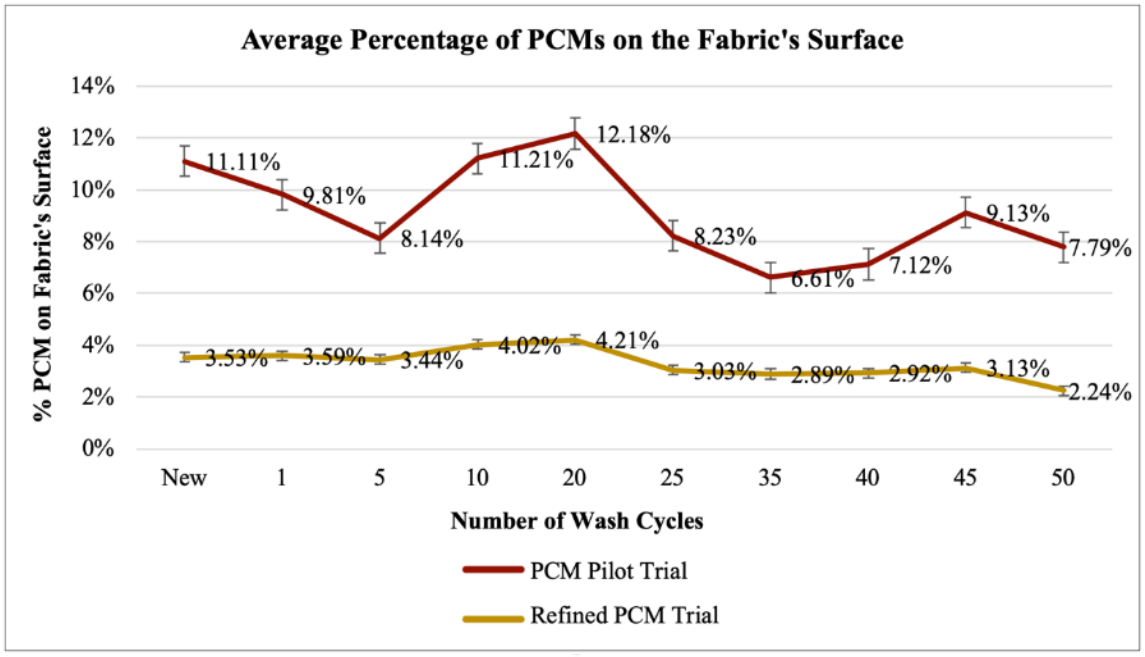


Figure 3. Graph comparing the fluctuations of data points between the initial pilot trial and the refined methodology of the second analysis trial.

5. Discussion and Conclusions

This research was conducted to develop and assess a new methodology for visualizing and quantifying microencapsulated PCMs on the surface of a fabric. The need for method development was based on current gaps in the literature for which an accessible, affordable, and reliable method for PCM fabric surface quantification does not exist.

The initial pilot analysis trial relied heavily on researcher inference to distinguish what was and what was not a PCM on the fabric's surface. As shown in Table 1, some clusters of PCMs were extremely recognizable, with clear boundaries and a visible distinction from the surrounding fibers. Others appeared blurry and it was hard to tell, in many cases, if they were a cluster of PCMs or if it was merely light reflecting off the synthetic, polyester fibers. This created a need to further refine the microscopic imagery methodology and analyze its efficacy in a subsequent analysis trial. Even so, the pilot trial of the newly developed methodology did successfully indicate a quantified reduction in the presence of the PCMs on the fabric's surface, over the course of the garment's wash life, which is in line with the visual microscopic images shown in Figure 1.

The second trial added standard criteria for clearly defining what constituted a PCM on the fabric's surface. The average percentage of PCMs on the fabric's surface were significantly lowered when using this analysis method compared to the results in the pilot trial. However, the fluctuations in the presence of the PCMs on the fabric's surface across the 50 laundering cycles followed a similar pattern to the pilot trial, but contained much less variability. This demonstrates that the refined method increased quantification accuracy for the PCMs that were visible on the surface of the fabric in the microscopic images.

The non-linear pattern of the results also sheds light on the way the PCM finish interacts with the fabric that it is printed upon during its wash life. Despite the images being taken in the same location, the differing appearances and visibility of the PCM indicates that the microcapsules move and migrate during laundering. This may be due to movement of the surrounding fibers, as well as, movement of the fibers the PCMs

are directly bonded to. Evaporation and abrasion of the PCM are other potential factors causing the shifting of the microcapsules during laundering. This movement could potentially account for the fluctuations in the data over multiple wash cycles.

While there were no statistically significant differences between wash intervals for either method, the quantified differences that were detected repeatedly within both methodology trials demonstrates the overall potential of the developed method for PCM fabric surface quantification. Overall, the developed methodology was able to demonstrate the decrease in the fabric surface presence of PCMs and effectively capture visual surface changes. This methodology may serve to provide researchers, product developers, and manufacturers with an accessible and affordable way to gather data and deepen understanding of a technology that is becoming increasingly prevalent in the retail market.

Limitations of the developed methodology still exist and therefore, this method should continue to be further refined and improved for its use as a simple, cost effective, and accessible way to visualize and quantify the change in the presence of PCMs on the surface of a textile over time and after laundering. One major limitation is that the images from the inspection microscope can only provide a two-dimensional snapshot of the PCMs present on the fabric's surface and is not a true representation of how many PCMs are present within the entire fabric structure at any given time. However, for the purposes of PCM performance, only those PCMs that are present on the surface of the fabric are relevant as contact with the skin is necessary in order for the phase change mechanism to be enabled. Therefore, the researchers believe the developed quantification and analysis methodology is relevant and appropriate, even with this limitation.

Future research should continue to explore an affordable way to easily quantify the amount of PCMs on a fabric's surface over a garment's useful wash life. Additional research should also explore the changing patterns and behavior of the PCM during the wash cycle. In order to do so, a simple detection methodology, such as the one developed here within, is necessary. Various microscopic techniques, software tools, other types of PCM finishes, and different textile substrates should be evaluated utilizing the methodology developed within this study to further determine its efficacy as an industry tool.

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