Specific testing for smart textiles

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12.1 Introduction

12.1.1 Markets and applications

Since their emergence, the smart textile technologies have received increasing attention from researchers all around the world as well as support and enthusiasm from users waiting to see and try the latest innovation. Even if, over the last decade, textiles have suffered from delocalization of the production and other economic restrictions, smart textiles have been able to show the customers high value products that have changed the perception people have of the textile industry.

Nowadays, the main sector of smart textiles that has resulted into commercial products has been directly related to wearable electronics and monitoring sensors for healthcare and sports performance (Dalsgaard & Sterrett, 2014). Despite an aging population, people want to stay active and mobile without being stereotyped as elderly due to the use of medical devices required for monitoring their health. These bulky devices have now been replaced by similar products, made entirely of textiles and which are directly integrated into clothing for an aesthetic and discreet look.

Similarly, in sports and leisure activities, sensors can provide feedback on the heartbeat and breathing rate or even muscle activities, which is vital information to optimize training (McGrath & Ní Scanaill, 2013).

Smart textiles are also widely used in the protection market, where the need for performance is often as important as the need for comfort (Tang & Po, 2007). By integrating heating or thermoregulating materials within the worker's uniform, it is already possible to reduce their bulkiness while improving the overall comfort (Scott, 2005).

More generally, smart textiles find applications in a large variety of sectors such as transport, home textiles, construction, and fashion. With expansive research directed towards new topics such as energy harvesting textiles, it is expected for the smart textiles market to continue thriving over the coming years.

12.1.2 Recent evolution of smart textiles

Several generations of smart textiles have succeeded one another over the years (Van Langenhove & Hertleer, 2004). Each new generation offers a higher level of integration. At first, the functional element was simply added at the last step of the assembly, i.e., after finishing. Even though it was easier to manufacture, the level of integration was poor and the wearer's comfort was low. The second generation of smart textiles has shown improvement by working directly at the level of the textile structure:

knitted, woven, etc. Finally the upcoming generation might be able to provide functionality through innovative fibers and yarns.

In 2011, the European Committee CEN TC 248 WG 38 gave the following definition of a smart textile material: "Functional textile material, which interacts actively with its environment, i.e., it responds or adapts to changes in the environment (CENTEXBEL, 2017)." However, it appeared that the social or common definition of a smart textile was much broader and required the creation of subcategories: passive smart textiles, active smart textiles and ultrasmart textiles (Singh, 2004).

Passive smart textiles can only sense their environment but are not able to interact in any way with it (Singh, 2004). An example of passive smart textiles is conductive fabrics or optical fiber embedded fabric. Their properties are constant in time and do not vary according to their surroundings. Active smart textiles, on the other hand, have the faculty to act both as a sensor and an actuator. Some examples are phase change materials (PCMs), shape memory polymers, electrically heated clothing, etc. Stimuli from their environment can be used to create a change in their properties.

The last category is directly in line with the definition given by the European Committee because ultra-smart textiles can sense, react, and adapt to their environment. They rely on the integration of electronic devices within the textile so they are able to detect stimuli, analyze them, and modify their structure to adapt to the stimuli. Musical jackets, intended for deaf people, which are able to vibrate and light up according to the music being played in real time, are an example of such ultrasmart systems.

In parallel, a particular attention has been brought onto the durability of the products in order to commercialize them. Not only must the performance be obtained, but it must also be maintained through use and cleaning to ensure the product has a decent lifetime.

12.1.3 Challenges and needs for new standards

These new concerns have raised the question of testing smart materials and developing evaluation methods. Due to the reactivity of smart textiles towards their environment and the perception of external stimuli, existing standard test methods may not be applicable. Any change in mechanical stress, light, humidity, or temperature can trigger the functionality within the textile and change radically the results of the measured parameter. Therefore, the need to develop new methods becomes inevitable. Several aspects of smart textiles must be addressed (Hertleer & Van Langenhove, 2015):

- · Characterizing the properties
- · Evaluating the performance
- · Assessing the durability

In order to do so, it is necessary to first identify and categorize the different types of smart textiles since they will not all have the same working principle neither the same requirements in terms of measured parameters.

12.1.4 Initiatives around the world

The current lack of objective test methods impedes the commercialization process for new smart textiles. Due to the importance of the matter, several research groups

and normalization organizations have launched projects in order to develop new standards. Among them, SUSTASMART (Supporting Standardization for Smart Textiles), a European research consortium including several international partners, has been working on this problem for over 2 years (CEN, 2011). The project ended in March 2014. In North America, the ASTM (American Society for Testing and Materials) holds special workshops to try and gather ideas and information from textile specialists (AATCC, 2016).

Even though an important step has already been accomplished, there is still a lot of work ahead. Every research center that has been working with smart textiles and has developed its own internal test methods must now come together in order to have some test methods officially recognized and approved by standards organizations.

12.2 Testing conductive textile materials

12.2.1 Introduction

Conductive materials are commonly used for smart textile applications since they have the capacity to act both as power transmission media and sensors. The electrical resistance is one the most important characteristics when it comes to conductive materials and the level of requirement varies according to the intended application.

Though the measurement of accurate electrical resistance on solid metals does not present any difficulties, the case of conductive fibers, yarns, or fabrics is more complex. The questions of repeatability of the measurements, number of specimens, values of the mechanical tension applied on the specimens, etc. have been studied by research groups worldwide (Šafářová & Grégr, 2010).

Indeed, most conductive fibers or yarns are produced by applying conductive materials such as silver, copper, polyaniline, or other conductive polymers on polymer fibers and yarns by surface coating or electroplating techniques. Because of the irregularity of the fiber and yarn surface, the process may not lead to fully homogeneous coating, and therefore, the resistivity of the yarns will depend on the location of the measurement contacts (Bashir et al., 2012). The same principle applies also to filaments and yarns containing conductive particles such as carbon. The random dispersion of these particles in the polymeric matrix and the possible formation of clusters can also create variations in the electrical resistance of the yarns (Smith, 2010).

Despite these obstacles, significant advances have been made during the last few years. Some existing standards such as ASTM D4496 (2013) or AATCC TM76 (2011) have been demonstrated to be applicable to measure the surface resistivity of fabrics. In parallel, the European research group Sustasmart has drafted a new standard proposal for measuring the electrical resistivity of yarns, which has been submitted to CEN (European Committee for standardization) and is pending approval (BS-EN-16812, 2016).

12.2.2 Electrical characteristics

Several parameters can be used to characterize the conductivity of materials:

- The electrical resistivity (ρ) only depends on the nature of the material and is constant regardless of its dimensions. It is often expressed in Ohms/m since the diameter of the yarn or cable is so small compared to its length that it becomes not significant. For fabrics, surface resistivity in Ohms/m² can be used.
- The electrical resistance (R) depends on the dimensions of the product (length (L), diameter (A)) and is expressed in Ohms. It is possible to compute the electrical resistance of a piece of material by using the following equation:

$$R = \rho * (L/A) \tag{12.1}$$

The equation demonstrates the existing relationship between the length of the cable and its electrical resistance. The longer the cable will be, the more the electrical resistance will increase. Contrarily, the larger the diameter of the cable will be, the more the electrical resistance will decrease, since the current will be able to pass more easily.

Conductance (G) and conductivity (γ) are respectively the inverse of the resistance and the
resistivity and are expressed in Siemens and Siemens/m.

Moreover, these parameters can be affected by external factors such as the temperature, compression, stretching, humidity, etc. In some cases, the effect of the environmental parameters on the electrical resistance is documented and can be computed. For example, the resistance will increase as the ambient temperature T decreases according to the following formula (Larrimore, 2005):

$$\rho_T = \rho_0 \left(1 + \alpha \left(T - T_0 \right) \right) \tag{12.2}$$

where ρ_T is the resistance at the chosen temperature and ρ_0 is the resistance at 25°C (at T_0).

The general trend for the effect of mechanical compression and stretching on conductive materials is also known: compression increases the contact between the conductive fibers and therefore decreases the resistance, whereas stretching increases it (Ding, Wang, & Wang, 2007). However, there is no mathematical relationship that describes the effect of mechanical compression and stretching on conductive material electrical characteristics since the hardness, elasticity, and other physical characteristics of the material will influence the result.

12.2.3 Measurement of the linear resistivity

The CEN committee TC 248/WG 31 on smart textiles standardization has been working on developing a testing method for measuring the linear resistivity of fibers, yarns, ribbons, and other materials for which the width of the conductive tracks is not significant compared to its length, i.e., $<10^{-1}$ (BS-EN-16812, 2016).

It is important to note that the width of the conductive track does not always correspond to the diameter of the conductive yarns. In the case of a knitted fabric for example, the width will be the loop height, whereas its length will correspond to its course, as illustrated in Fig. 12.1.

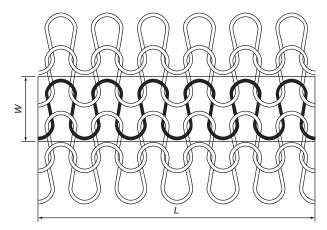


Fig. 12.1 Schematic representation of the width and length of the conductive track of a knitted fabric.

Reproduced with permission from CEN EN 16812.

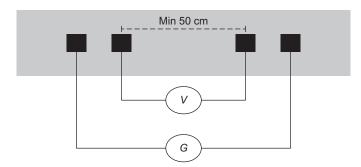


Fig. 12.2 Schematic representation of the test method set-up for measuring linear electrical resistance of conductive tracks.

Adapted from BS-EN-16812. (2016), Determination of the linear electrical resistance of conductive tracks. Brussels: CEN—European Committee for Standardization.

The test method uses four electrodes, connected to the conductive track and aligned over its width. The two inner electrodes are connected to a voltmeter, while the two outer electrodes are connected to a power source. The set-up is represented on Fig. 12.2, where *G* stands for power source and *V* stands for voltmeter.

The resistance (R) of the conductive material can then be calculated using the value of current (I) supplied by the power source and the voltage (V):

$$R = V / I$$

The electrodes must have a flat surface and provide a good contact with the conductive media. To improve this contact and depending on the nature of the conductive tracks, it may be possible to glue them with some conductive epoxy or solder them. In the case of conductive yarns, crimp connectors can also be used as electrodes.

Nature	Weight (g/m²)	Stress applied
Yarns	_	0.5 cN/tex
Not stretchable fabrics	<200	2 N
	200–500	5 N
	>500	10 N
Stretchable fabrics	_	0.5 N

Table 12.1 Values of stress to be applied on the yarn or fabric for the measurement of linear resistivity

The distance between the voltage electrodes should be at least 10 times the width of the conductive track or a minimum of 50 cm (Fig. 12.2).

A controlled value of stress is applied to the textile before the test. That value depends on the type and weight of the textile (Table 12.1).

The test method requires measuring at least five specimens. For each specimen, two series of five voltage measurements are done. Between the two series, the specimen should be relaxed and then retensioned.

12.2.4 Measurement of the surface resistance

Standardized test methods already exist for the measurement of the DC surface resistance of conductive materials. For instance, ASTM D4496 (2013) describes a testing protocol similar to the four points method used for the measurement of the linear resistivity (Section 12.2.3). The main difference consists in the weight and stress applied to the fabric as well as the position of the electrodes. For the measurement of the surface resistance, instead of having the four electrodes positioned on the same face of the conductive track, they are located on both sides of the fabric, as shown in Fig. 12.3.

Insulating materials can be added between the electrodes to support the fabric specimen and avoid the formation of a concave shape. A first weight is added above the current electrodes. It shall be $300\,\text{N/m}$ times the width of the specimen in meter. The second weight over the potential electrodes is $60\,\text{N/m}$ times the specimen width. The distance between the potential electrodes shall be superior to $10\,\text{mm}$, and they should be distant from the current electrodes by at least $20\,\text{mm}$.

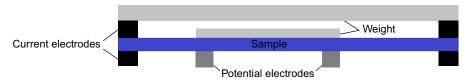


Fig. 12.3 Schematic representation of the electrode position for the measurement of the surface resistance of fabrics.

Adapted from ASTM D4496. (2013). Standard test method for D-C resistance or conductance of moderately conductive materials (5 pp.). West Conshohocken, PA: ASTM International USA.

The standard AATCC TM76 (2011) proposes a simplified method where the specimens are placed in contact with only two parallel plate or concentric ring electrodes that are distant by 25 mm. The electrodes are connected to an ohmmeter and a power source that is set to deliver 80 V for 1 min or as long as needed to reach a stable value. Two sets of three specimens are to be tested according this method. It is expected that each set will have a different orientation in terms of width and length. All specimens must be conditioned prior to testing; however, the values of temperature and humidity selected may depend on the intended application of the product.

12.2.5 Dynamic measurements

Most of the standards previously described impose controlled testing conditions so that a reference value of the electrical resistance can be accessed. However, electrical resistance depends on a number of environmental factors such as temperature, humidity, and mechanical stress. For instance, the electrical resistance may vary when the fabric is stretched, compressed, or abraded. In addition, that variation may not be the same if the fabric is a knitted or a woven structure.

Therefore, CTT Group has developed specific test methods to evaluate the change in electrical resistance when a mechanical action is applied on the conductive textile. For example, a setup allows evaluating the impact of abrasion on the electrical resistance of narrow woven fabrics comprising conductive yarns (CTT Group, 2011) (Fig. 12.4). It includes an electronic system to record the value of the electrical resistance every 20 abrasion cycles.

Data gathered from this test help determine the behavior and durability of the smart textile over time and under accelerated use conditions. Moreover, the conductive yarns can also be used as aging indicators to monitor the structural integrity of the fabric.

A system has also been developed to investigate the variation in electrical performance of smart fabrics subjected to tensile stress (CTT Group, 2010). As illustrated in Fig. 12.5, the conductive knitted fabric is held between the two clamps of a dynamometer while the resistance measurement is performed.





Fig. 12.4 Pictures of the recording device (left) and mechanical abrasion system (right) developed to evaluate the impact of abrasion on the electrical resistance of narrow woven fabrics comprising conductive yarns.



Fig. 12.5 Measurement of the electrical resistance of a conductive fabric under tensile stress.

12.3 Testing smart thermoregulating textiles

12.3.1 Introduction

The human body is designed in a way that allows it to regulate its internal temperature within a certain range, typically from 36°C to 38°C. In order to do so, several possible heat exchange mechanisms may take place (Arens & Zhang, 2006):

- Heat conduction happens through contact between the skin and another surface. If the surface is cooler than the skin, the body will evacuate heat, whereas if it is hotter, the heat will warm up the body.
- Thermal convection depends on the air flow around the body
- Heat radiation comes from infrared rays that reverberate on walls. The warm feeling of the sun behind a glass is an example of heat radiation.
- Sweat evaporation and breathing are other forms of heat transfer that are based on moisture transfer unlike the three others, which are dry exchanges.

However, in extreme climatic conditions such as arctic regions or deserts, the human body faces limitations and cannot provide enough heat or active cooling to keep his core temperature within an acceptable range. Therefore, clothing must act as a protective barrier between the body and his environment.

Thanks to emerging innovative textiles, it is now possible to integrate active functions within the fabric to compensate for the impact of the weather conditions. Several heating garments such as heated gloves, jackets, boots, etc. are already on the market (Wang et al., 2010). Cooling garments are more complex to produce and are still at an early stage of development despite some commercial examples. Developing thermoregulating materials that can provide independently both heat and cooling effects is a challenge that PCMs have not fully overcome yet (Mondal, 2008).

12.3.2 Conductive heating garments

Conductive heating garments come in various forms: nonwovens, knits, wovens, embroideries, etc. They all use the same principle to dissipate heat when they are powered: the Joule effect.

In order to measure how much heat is generated by the heating garment, thermocouples have been used to give a general idea of the textile temperature. However, results are generally not very precise/reproducible. Indeed, if the conductive element is dispersed randomly or inserted according a predetermined pattern, the choice of the position of the thermocouple on the heated surface will greatly impact the result obtained.

To measure a surface temperature rather than a point temperature, thermal infrared cameras are very useful and provide reliable data, generally within ± 1.5 °C, depending on the accuracy of the emissivity value used. A visual color gradation may be used to reveal the presence of hot points or colder areas as shown in Fig. 12.6.

In order to fully appreciate the color gradation along with the increase in dissipated heat, the range end points must be determined first. The distance between the heating textile and the camera may also have an impact on the results and should always be the same for comparative studies (Michalak, Felczak, & Więcek, 2009; Banaszczyka, Anca, & De Mey, 2009).

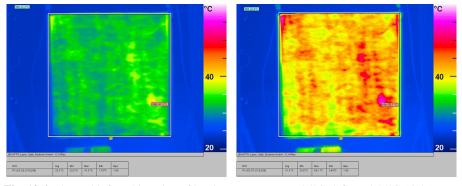


Fig. 12.6 Thermal infrared imaging of heating nonwovens at 35°C (left) and 45°C (right).

Knowing the average surface temperature of a heating element is essential but it is not sufficient to predict the behavior of the heating product while in use in a cold environment. Indeed, the time during which the heating garment will be able to provide heat is another critical element. In particular, low temperatures have a direct impact on the electrical resistance of conductive textiles, which will decrease and therefore require more power to maintain the same level of heating. It is possible to predict the electrical resistance of a heating textile at different temperatures if some characteristics of the conductive element are known (Larrimore, 2005):

$$\rho_T = \rho_0 \left(1 + \alpha \left(T - T_0 \right) \right) \tag{12.3}$$

Here, ρ_T and ρ_0 are respectively the resistance at temperature T and T_0 . ρ_0 is measured at ambient temperature (T_0) . α is a constant that depends on the nature and composition of the materials and refers to the temperature coefficient of the metallic element.

Using an empirical method is another alternative that proved to be sometimes more accurate than the theoretical model since it is closer to the real situation. For instance, heating garments are placed into a climatic chamber while powered, and their resistance is measured over a large range of temperatures.

Once the values of resistance and voltage (fixed) are known, the current can be computed. By dividing the energy contained in the battery (J) used to power the garment by the current (A), the time during which the heating garment will be operating can be calculated.

12.3.3 Cooling garments

Cooling garments are more complex and usually present a level of integration lower than the conductive heating textile. This is mainly due to the fact that current cooling garments are based on water or air circulating within small tubes inserted between two textile layers (Kim et al., 2011).

Currently there are only two existing standardized test methods that allow evaluating the performance of personal cooling system (PCS): ASTM F2371 (2011) and ASTM F2300 (2016). They both offer very different approaches: ASTM F2371 relies on the objective measurements performed on a thermal manikin, whereas ASTM F2300 monitors the physiological parameters of a panel of human subjects.

The standard test method ASTM F2371 (2011) evaluates two main aspects: heat removal rate and the duration of the cooling effect. The heated manikin used for this test shall be equipped with the sweating skin system since the cooling resulting from sweat evaporation represents a large fraction of the heat dissipation and must therefore be taken into account. The conditions specified for the test include an ambient temperature of 35°C with a relative humidity of 40% and an air velocity of 0.4 m/s.

A baseline test shall be conducted to isolate the effect of cooling only. If the PCS is active, the baseline test is conducted while the system is turned off. If the PCS is passive and cannot be turned off such as in the case of ice or gel vests, the garment is

placed into the climatic chamber for 12 h prior to the test in order to attain equilibrium between the PCS and the climatic conditions.

The manikin is dressed with clothes that are intended to be used with the PCS so the output is relevant to the real operating conditions.

The power input needed for the manikin to maintain a skin temperature of 35°C is recorded. An average of measurements carried out once steady-state conditions are reached is calculated and serves as reference for the PCS performance test.

The procedure is similar for the test. However, the PCS is turned on for that step. The manikin's skin temperature, the air temperature, and the power input to the manikin are recorded continuously for 2h or until the difference between the power input during the test and that corresponding to the baseline test has reached 50 W. It is expected that the power input to the manikin with the PCS turned on is greater than for the baseline test since the PCS should attempt to decrease the skin temperature: Additional heat is thus required to maintain the manikin temperature at 35°C.

Even though only one sample of the PCS is used to conduct the test, the measurement shall be replicated three times to take into account variations in dressing and instrumentations.

The heat removal rate is calculated by dividing the average power input (PCS test minus baseline test) by the cooling duration which is the time it took to reach a difference of 50W.

The standard ASTM F2300 (2016) may be an interesting alternative if no thermal manikin is available. However, it requires more logistics and time and is more costly as it involves human subjects. In addition, less flexibility is allowed for the under and outer garments worn by the subjects during the test than in the method ASTM F2371. At least five human subjects are required to evaluate the performance of one PCS. Physiological parameters include esophageal, rectal, and skin temperature as well as heart rate and body sweat rate. A treadmill is used to help participants generate a sufficient metabolic energy expenditure that will translate in a temperature rise. The duration of the cooling can be deduced from the time needed for the core temperature to reach 39°C or for the skin temperature to reach 38°C. If none of these two happens within 2h, the test is stopped.

The test method described by ASTM F2300 provides more details than in ASTM F2371, but is also more complex to interpret and relate directly to the PCS performance. Moreover, it involves much more subjectivity than with the use of an instrumented manikin.

12.3.4 The specific case of PCMs

PCMs have the unique feature of being able to either provide heat or cool down (Mondal, 2008). They are, however, restricted in terms of the duration of this effect. Indeed, they are active only while they are at the transition between the liquid and solid phases (from liquid to solid for a heating effect or the opposite for a cooling effect). In order to evaluate the performance of PCM-based textiles, several methods can be used. The most common is the differential scanning calorimeter (DSC), which is used to measure the phase transition range as well as the enthalpy of both the melting

and the crystallizing process. However, this method is limited by the fact that it does not take into account the thermal barrier effect of the PCM (Sharma et al., 2009).

In 2002, Hittle & Andre developed a test method that was standardized in 2004 as ASTM D7024 (withdrawn, 2013). It is based on the use of an index called the temperature regulating factor (TRF), which is computed using the following equation (ASTM D7024, 2013):

$$TRF = \frac{\left(T_{\text{max}} - T_{\text{min}}\right)}{\left(Q_{\text{max}} - Q_{\text{min}}\right)} \times \frac{1}{R}$$

where the amplitude of the temperature variation $(T_{\rm max}-T_{\rm min})$ is divided by the heat flux variation $(Q_{\rm max}-Q_{\rm min})$ and the thermal resistance of the fabric tested. The TRF is a dimensionless number lower or equal to 1.

A specimen of the tested fabric is placed between a hot plate and two cold plates, one on each side of the hot plate/fabric system (Fig. 12.7). The cold plates are maintained at 20°C, whereas the hot plate is subjected to a sinusoidal flux. The midpoint of the sinusoidal flux is 150 W/m² and its amplitude is 100 W/m² above and below the midpoint. The thermal resistance value is measured under steady-state conditions when the hot plate is submitted to a constant heat flux of 250 W/m².

Hittle and Andre have pursued their work in order to take into account other parameters than the latent heat: diameter of the PCMs, amount of PCMs in the fabric, method of incorporation of the PCMs, etc. Another test apparatus has been developed for this purpose and is called the fabric intelligent hand tester (FIHT) (Ying et al., 2004). It is composed of a bottom measuring plate and an upper measuring head which integrates temperature, pressure friction, displacement, and heat flux sensors.

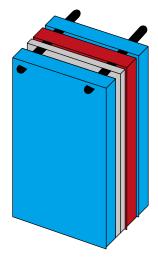


Fig. 12.7 Schematic illustration of the TRF test apparatus with the cold plates in *blue*, the hot plate in *red*, and the test specimen in *gray*.

Several indexes may be calculated using the data collected in order to analyze the performance of the PCMs in a broader way: static thermal insulation (I_s), phase change duration time (Δt_d), PCM heat flux during the phase change period (I_d), and thermal psychosensory intensity (TPI).

More recently, studies have turned towards the guarded hot plate system (ISO 11092, 1993), which is commonly used to determine the static thermal resistance of fabrics, to evaluate the PCM impact on the heat flux variation (Wan & Fan, 2009; Bendkowska & Wrzosek, 2009). In particular, the use of already existing standardized methods is beneficial since they allow comparing passive and active materials.

12.4 Testing ECG, EMG, EEG, and other embedded textile sensors

12.4.1 Introduction

Over the past few years, the healthcare market has invested more than 3 billion dollars in textile materials and research (Dalsgaard & Sterrett, 2014). The medical sector is constantly looking for new technologies that will facilitate the continuous monitoring of patients at risk while not impeding their lifestyle. Smart clothing offer the advantage of direct and permanent contact with the body, allowing access to bioparameters such as ECG (electrocardiogram), EMG (electromyogram), EEG (electroencephalogram), breathing rate and others.

Driven by the need for such products, several companies have developed shirts, chest-belts, and bras that integrate electrodes capable of detected biosignals and transferring them to a data acquisition module. Companies such as Ambiotex, OM Signal, Under Armour, etc., have already made commercial products available on the market. Among them, Zoll Life Vest went even further by embedding not only electrodes but also a defibrillator in a garment (Francis & Reek, 2014). It is programmed to trigger an electric discharge in case of a heart failure.

This last example demonstrates the need to ensure high reliability, accuracy, and robustness of biomonitoring clothing. Due to the high demand context of the application, the tolerance for false signal, lack of thereof, or even drifting is very low. Therefore, biomonitoring garments shall be carefully tested before their market entry as well as on a regular basis during service.

12.4.2 Evaluation of electrode adhesion

ECG measurements are based on the detection of the depolarization of the ventricle when it contracts. The electrical pulse that accompanies the depolarization is channeled through the electrodes to create the signal (Becker, 2006).

In order to reduce the skin-electrode impedance and avoid motion-induced artefacts, the electrodes must perfectly adhere to the skin. Traditional electrodes use adhesive conductive gel to improve the contact at the interface. However, if they are worn during extended periods of time, these electrodes may create skin irritation

(Merritt, 2008). The adhesion strength of this type of electrodes is measured with a standard peel test.

Textile electrodes do not comprise any form of adhesive layer since, thanks to its compressive properties, the base fabric induce the contact with the skin. In this context, peel tests are not relevant.

The measurement of the coefficient of friction of the electrode can, however, help determining its surface properties and identifying if the electrode would tend to slide. The traditional friction test is generally based on the use of a sled over which is positioned the tested material, and which slides over a substrate. The coefficient of friction μ is given by the following equation (Blau, 2001):

$$\mu = f/N \tag{12.4}$$

where f represents the friction force and N is the normal force exerted by the sled. Therefore, the tightness of the shirt around the chest can be taken into account and described through the value of N. Ideally, a material having surface properties similar to skin should be chosen as the substrate on which the sled bearing the electrodes will be slid to determine the coefficient of friction.

Two parameters must be considered: the static coefficient of friction and the dynamic coefficient of friction. The static friction coefficient corresponds to the force necessary to initiate a motion of the upper material (i.e., the electrodes) against the substrate (i.e., the skin analog). The dynamic coefficient of friction is given by the force required to maintain the motion (De Luca et al., 2004).

The standard test method ASTM D1894 (2014) for plastic films and sheeting is commonly used in the textile industry and recommends a displacement speed of $150\pm30\,\text{mm/min}$. The weight applied on the sled should correspond to the level of compression of the garment over the body.

12.4.3 Measurement of the electrical impedance

The electrical impedance between the skin and the electrode is one of the most determining factors of the signal quality. Indeed, a high value of impedance will induce a small signal/noise ratio and require that the signal is strongly amplified and filtered before being usable.

An equivalent electrical circuit can be established for the skin-electrode impedance. For instance, Swanson and Webster have proposed a model that consists of a resistor (R_e) in parallel with a capacitor (C_e), associated with a second resistor (R_s) in series, as shown in Fig. 12.8 (Assambo et al., 2007; Taji et al., 2014). The skin-electrode impedance can be deduced from measurements of the electrical characteristics of the electrodes and the skin.

Despite the fact that this method is easy to implement, it does not provide very accurate values of the skin-electrode impedance. Indeed, the effect of the body fluid motion and skin surface humidity has to be taken into consideration. To do so, Priniotakis et al. (2007) have used electrochemical impedance spectroscopy to develop an electrochemical cell (Fig. 12.9) with the objective of replicating the contact of the electrodes against the body with the influence of its fluids.

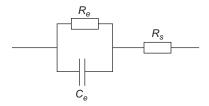


Fig. 12.8 Swanson and Webster single-time constant model for the skin-electrode impedance.

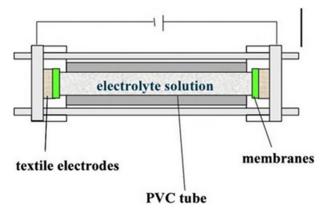


Fig. 12.9 Electrochemical cell developed to characterize the skin-textile electrode impedance. Reproduced from Priniotakis, G., et al. (2007). Electrochemical impedance spectroscopy as an objective method for characterization of textile electrodes. *Transactions of the Institute of Measurement and Control*, 29(3–4), 271–281, with authorization.

The cell is composed of a PVC tube filled with an electrolyte solution replicating the body fluids, two semipermeable membranes on both extremities of the tube, and the two electrodes on the external face of the membranes. The semipermeable membranes simulate the skin. Since skin pore size and density vary from one person to another, different membranes should be used for the test to take into account the variability in human skin properties. In the Priniotakis et al. (2007) study, two membranes were tested with 5 and 0.45 μm pore size.

It is recommended that several textile electrodes are tested to calibrate the electrochemical cell. Indeed, the surface profile of a knitted structure versus a nonwoven one is very different. Because textile structures form a complex network of point contacts with the porous membrane, the impedance cannot be obtained from the global contact surface but is provided by the sum of each individual contact.

12.4.4 Assessment of the sweating impact

The presence of humidity between the skin and the electrode will generate a decrease in the bio-impedance. As the textile electrode gets soaked by the liquid, the charges are able to move more freely through the system, resulting in a lower electrical resistance.

In addition, due to the presence of salts in human sweat, it is expected that the electrical resistance drops even faster than with tap water (Brahme, 2014).

Westbroek et al. have conducted a study on the short- and long-term evolution of the electrical resistance of textile electrodes after exposure to sweat (Westbroek et al., 2006). The electrical resistance of nonwoven electrodes was reduced by 20% just a few minutes after being exposed to synthetic sweat. Lower variations were observed for knitted and woven electrodes. This was attributed to their lower liquid absorption capacity than nonwovens.

Regarding long term exposure, the electrical resistance of all the electrodes had increased by 25% after 4 days and by more than 40% after 20 days (Westbroek et al., 2006). This was attributed to the effect of corrosion. In this experiment, the synthetic sweat solution was produced using 20 g/L of NaCl, 1 g/L of urea, 500 mg/L of salts, and the addition of NaoH or HCl to reach a pH of 5.8.

Standardized recipes for synthetic sweat preparation are available in ISO 11641 (2012), ASTM D4265 (2014), and AATCC TM15 (2002). Artificial perspiration may be acidic or alkaline. For instance, ISO 11641 (2012) recommends reaching a pH of 5.5 ± 0.2 for an acidic solution and 8.0 ± 0.1 for an alkaline solution. In all cases, NaCl remains the most important component of the solution regardless of the pH.

Since the corrosion reaction is mostly due to the presence of chloride, an increase of its concentration in the solution can induce an acceleration of the degradation of the conductive fibers. Therefore, this method can be used to simulate the ageing of the textile electrodes and conclude on their durability.

12.4.5 Assessment of the comfort properties for long-term use

Textile electrodes are preferred to conventional electrodes for long-term monitoring because they can be integrated into clothing and are usually more comfortable to wear for extended period of time.

An important requirement for textile electrodes is that the electrode surface is non-reactive with skin. This property is typically obtained with the use of silver plated yarns that often combine high electrical conductivity and biocompatibility properties.

In order to assess the comfortable feeling associated with wearing the electrodes, two modes of evaluation can be considered:

- The comfort wear test is a subjective evaluation that involves several participants grading the garment based on scales from 1 to 5 while performing specific activities (box lifting, ladder climbing, etc.) (Hollies et al., 1979). The ASTM F1154 (2011) method provides an exercise scenario for the assessment of chemical protective garments, but it may be applied to other clothing items. Although the testing procedure is close to the intended use of the garment, this method of evaluation remains costly and time consuming.
- The Kawabata evaluation method uses a series of 4 instruments to characterize, in an objective
 way, the fabric properties, including the tensile strength and shear stiffness (KES-FB1), bending
 rigidity (KES-FB2), compression (KES-FB3), and surface friction and roughness (KES-FB4).

In the context of textile electrodes, the measurement of the coefficient of friction and surface roughness is the most relevant parameter to consider since the electrodes will be integrated within a piece of clothing. Therefore, properties such as bending rigidity, compression, etc. should be directly tested on the supporting fabric because the contribution of the electrodes generally remains negligible.

The KES-FB4 uses 10 sensors to simulate the fingertip feeling and records the output required to move the fabric at a constant rate of 0.1 cm/s over a distance of 2 cm (Barker, 2002). Simultaneously, a probe is placed on the fabric during its displacement to measure the mounts and valleys, i.e., the roughness of the surface.

The friction coefficient provided by the KES-FB4 ranges between 0 and 1, with 1 representing a very sticky surface. The surface roughness expressed by a number between 0 and 20, where 20 represents a very coarsely-textured fabric.

12.5 Testing cosmetotextiles and smart dermotextiles

12.5.1 Introduction

Due to their close contact with the body, textiles represent ideal substrates to carry and deliver cosmetic products to the skin. The active principles are encapsulated within microspheres which are, then, dispersed either inside the fibers or in a coating paste. Because of friction, chemical reaction with sweat, or change in pH, the microcapsules degrade as time goes on. Upon degradation, they release their active ingredient at a constant and regular rate (Persico & Carfagna, 2013). That rate is controlled by the thickness of the polymeric capsules.

The same principle is also used in medical applications for the controlled release of drugs. In the case of cosmetotextiles or dermotextiles, properties such as moisturizing, slimming, energizing, etc. are targeted and are integrated into pantyhose, underwear, or socks.

Several commercials products have already been launched on the market despite the absence of standards to evaluate their actual performance. Because of the variety of compounds encapsulated as well as their intimate contact with the skin, cosmetotextiles should not be evaluated only for their effectiveness but also in regards with their eventual toxicity and their durability after washing cycles. Finally, specific labeling must be put in place in order to subcategorize products and inform consumers.

12.5.2 Evaluation of the chemical properties of the active ingredient

Before evaluating the efficiency of the cosmetotextiles, the chemical components shall be characterized and quantified. Each ingredient (copper oxide, chitosan, aloe vera, etc.) shall be tested and identified according to specific test methods. For example, the presence of vitamin E can be detected by dripping FeCl₃- onto the finished textile (Singh, Varun, & Behera, 2011). The ions Fe²⁺ created by the reaction are evidenced by immersing the textile in a solution of Dipyridyl where the ions Fe²⁺ react to create a red complex which is visible to the naked eye.

The component analysis of perfumed textiles is more complex and requires headspace gas chromatography and mass spectrometry because of the volatile nature of these compounds (Barel, Paye, & Maibach, 2014). The specimen is placed in a vessel under vacuum and is submitted to temperature variations. The vapors are collected and analyzed for odor issues, identification of polymer additives, or residual solvent. Testing protocols may be found in ASTM D3362 (2005), ASTM D3452 (2012), or ASTM D4128 (2012) for instance.

12.5.3 Evaluation of the efficiency of the claimed effect

Commercially available cosmetotextile products usually claim a wide variety of actions ranging from slimming and moisturizing to energizing and perfuming effects. Since 2009, the European directive 76/768/CEE requires manufacturers of such products to provide evidences of the effects advertised to the competent authorities (Bartels, 2011). In order to do so, four main methods are commonly used (Colipa—European Cosmetics Association, 2008):

- In vivo sensorial testing refers to a visual, tactile, or olfactory evaluation of the product effect by consumers or human subjects. This method is often used for perfuming or deodorizing textiles.
- In vivo instrumental testing also requires the involvement of human subjects. However, in this case, objective criteria or skin properties are measured by bioengineering techniques.
- Ex in vivo instrumental testing is very similar to in vivo instrumental testing. The only difference lies on the fact that the testing does not take place on human subjects but on specific human elements such as hair braids, skin tape strips, etc.
- In vitro testing uses artificial media recreated in laboratory in order to perform the tests.

For example, conductimetry or corneometry measurements may be conducted on human subjects before and after wearing moisturizing cosmetotextiles to determine the efficiency of the product. This technique relies on the electrical conductivity of the outer skin layers, which will directly be modified by any change in hydration.

Another example would be the evaluation of slimming textiles that can be achieved by the quantification of cellulite by echography of the dermal density. Similarly, the firmness and elasticity of the skin can be measured either by cutometry or ballistometry. A negative pressure is induced so that skin is locally sucked up by the instrument while the amplitude and duration of the deformation is noted.

12.5.4 Toxicity and innocuousness

Because of their close contact with the skin, cosmetotextiles may lead to risks of irritation and even toxicity. Indeed, most of cosmetotextiles use a microencapsulation process to contain the active substance. Therefore, chemical agents involved in the production of these microcapsules stay on the skin long after the microcapsules are empty and may lead to skin sensitization or epidermal cell toxicity.

In order to identify these potential negative outcomes, cosmetotextiles should be submitted to testing by either one of those three standards (Barel et al., 2014):

- EN ISO 10993 (ISO 10993, 2009) for medical devices includes two parts of interest for cosmetotextiles and smart dermotextiles:
 - Part 5: Tests for in vitro cytotoxicity

- Part 10: Tests for irritation and skin sensitization. The method involves both in vitro and
 in vivo testing. The in vitro procedure uses rat skin and human skin cells, whereas rabbits
 are used for the in vivo tests. A scale of 0–4 allows the grading of any intracutaneous
 reactions.
- OECD guidelines for testing of chemicals, section 406 (1992), describes a test method for skin sensitization that is inspired from the guinea pig maximization test (GPMT) by Magnusson and Kligman (Kligman & Basketter, 1995).
- OEKO-TEX is an association of several Institutes of textile research and testing in Europe
 and Japan that offers a certification for textiles tested for various harmful substances such as
 heavy metals, formaldehyde, etc. (Niinimäki, 2006). Among the list of agents tested, solvent
 and other chemical residues would be of interest for cosmetotextiles.

12.5.5 Durability

When it comes to the durability of cosmetotextiles, two aspects should be considered independently: the durability of the active ingredient and the durability of the microcapsules. The release mechanism of the active substance is timed to last a determined number of hours, days, or weeks depending on the intended use. Unless the microcapsules are faulty, the product should be able to deliver the cosmetic effect over the planned period of time.

However, other parameters such as rubbing, temperature change, the presence of sweat, etc. can affect the performance of the binder used to create the adhesion between the microcapsules and the textile, leading to their detachment and loss. In order to verify the presence of the microcapsules on the fabric, the scanning electron microscope (SEM) and transmission electron microscope (TEM) have proven to be useful (Rodrigues et al., 2008; Monllor et al., 2010). Although it is difficult to quantify accurately the microcapsules with microscope imaging, the images provide

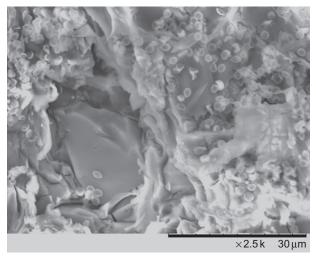


Fig. 12.10 Example of SEM image of microcapsules on a fabric.

a general estimate of the microcapsule concentration as illustrated in Fig. 12.10. Moreover, TEM can be used to identify if the microcapsules present on the fibers still contain the active ingredient or not.

Other techniques may be used to evaluate the residual amount of active ingredient inside the microcapsules. These techniques generally follow two main steps: extraction and quantification (Bartels, 2011). Solvent extraction is most commonly used but, in specific cases, the Soxhlet extractor may also be an option. The obtained solution is then analyzed by GC (Gas chromatography) or HPLC (high performance liquid chromatography) to separate and identify the components.

Azizi et al. reported using UV-visible absorption spectroscopy combined with solvent extraction to quantify the remains of neroline, a perfume component, after several washing cycles (Azizi, Chevalier, & Majdoub, 2014). Diethyl ether was used for the extraction and its UV spectrum had been checked against the UV spectrum of the extracted solution to identify the absorption maxima that would be characteristic of the neroline. The variation in the absorbance ratio was expressed as a function of the number of washing cycles.

12.5.6 Labeling

The European standard CEN/TR 15917 (CEN, 2009) differentiates product labels and the commercialization labels. Except for small articles such as socks or hoses, the product label shall be attached to the textile item, whereas commercialization labels are generally affixed on the package.

The product label shall display at least the following information:

- Fiber composition, based on the European directive 96/74/CE related to textile denomination:
- Care conditions in accordance with EN ISO 3758 (ISO 3758, 2012) "Textiles—Care labeling code using symbols";
- Tracking number (for example, batch or serial number);
- · Identification of the cosmetic product.

In Canada, cosmetic product labels shall list all ingredients in descending order of predominance down to the minimum weight of 1% (Canada Health, 2009)

Commercialization labels shall clearly state the following elements:

- The word "cosmetotextile" must be explicitly written.
- All cosmetic ingredients shall be listed in accordance with Article 6 of the European Directive 76/768/CE related to cosmetic products. Products may also be referred to by their INCI (International Nomenclature for Cosmetic Ingredients) code.
- It shall be recommended to keep this information with the product for its entire lifetime.
- The name and address of the manufacturer head office shall be indicated, although abbreviations are allowed.
- An expiry date shall be mentioned if the optimal period of usage is <30 months.
- Any particular precautions for use shall be stated if necessary.
- The main function of the product has to be written on the label unless its representation is clear enough.

12.6 Conclusion

Although there has been a lot of activity in the field of smart textiles standardization, no actual standard has still been approved and is officially being used by the textile industry. Looking back over the last decade, Europe has, so far, been the most proactive region for standard development for smart textiles. In 2006, they created a first task group that led to the well-known project "Sustasmart." Even though the project officially ended in March 2014, work continues: a first draft of the standard proposal titled, "Determination of the linear electrical resistance of conductive track" was submitted in 2015. It is expected to become the first official standard applicable for smart textiles.

In North America, interest for the development of new standards related to the emerging sector of smart textiles increased in 2012 when ASTM organized the first smart textiles workshop for standardization. More recently, a subcommittee of ASTM D13 on textiles dedicated to smart textiles, ASTM D13-50, was created.

In addition, the AATCC organization joined forces with ASTM and organized a joint workshop in March 2016.

It is worth mentioning the importance that people from the electronics sector are represented at these workshops and remain involved in the standardization process. Indeed, smart textiles usually require at least one electronic component or device, for instance a power source. The impact of the electronic part on the product performance can be crucial and should be included in the aspects evaluated by the standards.

In parallel, a number of research centers are developing internal test methods in order to be able to characterize and evaluate the performance of the products they develop and prototype. A large amount of knowledge has thus already been acquired on the topic. However, it lacks homogeneity, objectivity, and comparable basis. For instance, different methods can be used to test the same function, such as a heating textile for example, based on the equipment available: thermal infrared camera, thermocouple, etc.

The involvement of standardization organizations can help turn these various internal methods into a unique objective method that would become the reference for both the industry and the consumers.

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