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EVALUATION OF GRAPHENE WASHING FASTNESS ON CELLULOSE FABRIC AND METHOD TO IMPROVE IT

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Abstract: *Graphene is a recently discovered material that has attracted a great deal of attention in numerous fields, from electronics to textiles, including medicine and energy harvesting. Its use confers interesting properties to conventional textile substrates, providing them with great versatility and the opportunity to explore new areas and applications beyond their common uses. Despite the many properties it shows, it also faces some challenges regarding critical issues such as its stability throughout the procedures the textile is subjected to, such as rubbing, ironing or washing. In this study, the washing fastness of a cellulosic textile substrate to which a printing paste containing graphene had previously been applied was evaluated. After evaluating the behaviour of the samples after the washings by means of chromatic coordinates and the ISO 105:C06 Color fastness to domestic and commercial laundering, it came to our attention the deficient behaviour of the graphene regarding washing fastness. In order to improve it, a heat treatment was applied, which led to a significant increase of washing fastness behaviour of the samples with graphene printing, compared to non-treated samples. In this research, an efficient and affordable method to improve graphene's stability onto cellulosic textile substrates that can be carried out with common laboratory equipment is provided.*

Key words: *cotton, printing, thermal, luminosity, color, coordinates*

1. INTRODUCTION

Graphene is the name given to a flat monolayer of carbon atoms tightly packed into a two-dimensional (2-D) honeycomb lattice, first isolated in 2004. Graphene exhibits high electron and hole mobility, high thermal conductivity, as well as other features such as extremely high tensile strength, flexibility, stretchability and superior radiation hardness [1-3].

Possible applications of graphene materials include: flexible electronics, photonics and optoelectronics, spintronics, composite materials, energy generation and storage, biomedical applications, sensors, etc. [4-11].

There are four basic methods used for graphene synthesis: chemical vapor deposition; epitaxial growth of graphene on electrically insulating substrates; mechanical exfoliation of graphene from bulk graphite and reduction of graphene derivatives such as graphene oxide [12].

Mainly, three methods have been developed to produce graphene-based fabrics/yarns. The first one is the coating of fabrics/yarns with graphene materials such as graphene, graphene oxide, educed graphene oxide, etc. The second one consists in the chemical vapor deposition process of graphene on a metallic mesh (Cu normally) that is later removed by an acid treatment, remaining the graphene-fabric structure, these types of fabrics are named graphene woven fabrics. And the third one, includes the production of graphene fibers and its application on fabrics [4].

In the past few years, graphene applications within textile industry have gained researchers' attention and keep growing in number each year. Some examples of its usefulness comprise cotton fabric as flexible strain sensor based on hot press reduced graphene oxide [13], graphene nanoribbon coated flexible and conductive cotton fabric [14] and hydrophobic cotton textile surfaces using an amphiphilic graphene oxide coating [15] amongst others [16-21].

Nevertheless, it needs to be taken into consideration that textiles need to be able to undergo intense procedures such as washing or drying and keep their properties reasonably intact in order to last all their service life with a proper performance. Even though this is a critical issue to ensure graphene's industrial usage expansion, few researches have been focused on improving graphene's stability onto textile substrates so the lack of investigation within this area led us to develop an easily reproducible method to improve washing fastness on cellulose fabric.

2. MATERIALS AND METHODS

2.1 Fabric

For this research, a 100% cotton fabric with a grammage of 210 g/m2 and chemically bleached in an industrial process was used.

2.2 Chemicals

Graphene was supplied by Innovatec SC, S.L.

To obtain the printing paste, the following products were used:

- Lutexal CSN and Luprintol SE, supplied by Archroma
- Resina Center BC, supplied by Color-Center S.A.
- Ammonia 28%, supplied by Prolabo.

2.3 Procedure for graphene deposition onto fabric

Graphene deposition onto fabric was carried out by means of hand printing using the following recipe:

Product	Quantity (g)
Lutexal CSN	20
Resina Center BC	10
Luprintol SE	10
Ammonia	10
Distilled water	950
Graphene	12

Table 1: *Printing paste composition*

2.4 Washing test materials and procedure

Washing fastness was evaluated by a laundry according to the standard ISO 105:C06 for 30 minutes at 40º C. Treated sample was conveniently covered by a multifibre fabric.

2.5 Thermal treatment

Graphene-printed fabric samples were oven-dried at 100ºC until completely dried.

In order to improve washing fastness of graphene, a thermal treatment was applied to certain fabric samples for 3 minutes at 150ºC.

2.6 Color coordinate evaluation

In order to objectively compare color difference measurements, the chromatic coordinates (CIE L *, a *, b *) of the CIELAB color space of the printed samples were obtained using a MINOLITA CM-3600d reflection spectrophotometer. The measurements were made with the standard observer CIE-Lab 10^0 and the standard illuminant D65. According to ISO 105 J01:2000 Textiles -- Tests for color fastness -- Part J01: General principles for measurement of surface color

On the other hand, the color difference of the samples was obtained according to the following equation:

Color difference (ΔE*) = ((ΔL*)2 + (Δa*)2 + (Δb*)2)1/2

Where ΔL * = L * non treated fabric - L * treated fabric; Δa * = a * non treated fabric - a * treated fabric; Δb * = b * non treated fabric - b * treated fabric; "L *" describes the luminosity, "a *" measure of red-green hues, "b *" measure of blue-yellow shades. It should be noted that three measurements were made for each sample and the mean value was calculated.

3. RESULTS AND DISCUSSIONS

3.1. Visual comparison

Samples obtained after printing showed a considerable change in color as they were completely black**.**

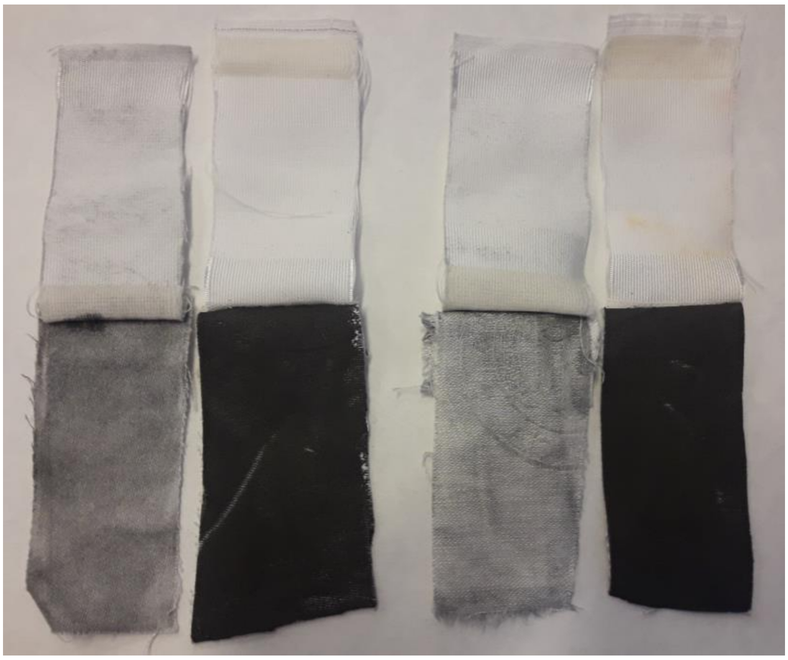

Fig. 1: *(From left to right) EG12-1-1000 without thermal treatment washed, EG12-1-1000 with thermal treatment washed, EG12-2-1000 without thermal treatment washed, EG12-2-1000 with thermal treatment washed.*

At a glance, the contrast between treated and non-treated samples is quite noticeable, as it can be observed in figure 1, where it seems quite evident that color of thermal treated samples remains almost intact, while non-treated samples lose a large amount of the stamping paste, thus reducing efficiency of graphene properties.

Not only have the non-treated samples lost more graphene than the treated ones, but also an important difference in the discharge towards the control fabric has been detected between them, with non-treated samples staining the control fabric slightly more than thermal treated samples.

3.2. Color coordinates

The chromatic coordinates obtained for each of the samples and the color differences are given in the following chart, where the abbreviation TT stands for thermal treatment and W for washed.

Sample	L*	a*	b*	DL*	Da*	Db*	DE*ab
Original fabric	93,228	-0,1247	4,406				
EG12-1-1000	29,6818	0,082	0,0536	-63,5462	0,2067	-4,3524	63,6954
EG12-1-1000– W	54,4692	-0,3646	-0,0455	-38,7588	-0,2399	-4,4515	39,0143
EG12-1-1000– TT – W	30,2406	0,0408	0,0188	-62,9873	0,1654	-4,3872	63,1402
EG12-2-1000	30,5858	0,0087	0,1039	-62,6422	0,1334	-4,302	62,7899
EG12-2-1000– W	67,5212	-0,5945	1,4332	-25,7067	-0,4698	-2,9728	25,8823
EG12-2-1000– TT – W	30,5086	0,0291	-0,1562	-62,7194	0,1538	-4,5621	62,8853

Table 2: *Chromatic coordinates for examined samples*

As it could be expected changes are appreciated only when analysing L* coordinate, and a* and b* remain practically constant. Thus, we can affirm that DE*ab is mainly due to the variation on L*. Checking the results obtained in the measurement of color, an important variation in the value of the luminosity is observed. Thermal-treated samples hardly show a difference in luminosity after washing, while in untreated samples this value increases remarkably, which indicates they are close to the value of the original tissue without stamping, having lost much of the deposited graphene.

3.3. Washing fastness evaluation

Once the samples had been washed and dried, it was compulsory to evaluate them considering both the color degradation (fade) and the stain on multifibre fabric.

Sample		WOOL	ACRILIC	POLYESTER	POLYAMIDE	COTTON	ACETATE
EG12-1	STAIN	4	4	4-5	4	4	4
	FADE	1-2					
EG12-1 THERMAL	STAIN	5	5	5	5	5	5
	FADE	4-5					
EG12-2	STAIN	4	4	4-5	4	4	4
	FADE	1-2					
EG12-1 THERMAL	STAIN	5	5	5	5	5	5
	FADE	4-5					

Table 3: *Color fastness to washing for tested samples*

According to the color fastness values, it is clearly observed the thermal treatment practically does not allow to miss graphene particles during the laundry, so a better performance of graphene is expected in this case; whereas samples without thermal treatment have missed a wide quantity of the printed graphene, consequently reducing the efficiency of properties given to the textile substrate by the graphene.

Moreover, as there is no affinity between graphene and the fibres, the stain on the multifibre fabric is not far away from the 5 value in non-treated samples but it is better in thermal treated samples, given the fact that there is almost no graphene miss that could stain the control tissue.

4. CONCLUSIONS

The main conclusion deduced from this research is that, when the proper amount of heat is applied, the printing paste cures, thus binding the graphene to the textile substrate. This leads to an outstanding improvement of washing fastness of the thermal treated samples.

Furthermore, being able to preserve almost all of the graphene applied to the textile substrate implies a better conservation of the properties provided by graphene deposition, such as its influence on thermal and electrical conductivity.

This research article demonstrates a fast, affordable and easily reproducible method to improve washing fastness of cellulose textiles with graphene, using common laboratory equipment, so it can be used at an industrial level, increasing graphene suitability for textile applications and its commercial worthiness.

In summary, it can be said that this study will expand the scope of graphene and open new research lines about its applications on cellulose fabrics. Future studies will be focused on the temperature and time optimisation.

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