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Innovation of Medical Protective Clothes Depending on Antiviral and Antibacterial Treatments

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Abstract: Research of antiviral textiles has received considerable attention owing to the continuous emergence of new infectious diseases. Antiviral textiles can effectively inhibit the spread of viruses and significantly reduce the risk of cross-infection and re-infection to protect people's health and safety. In recent years, researchers studied various antiviral materials, which can prevent the spread and reproduction of viruses by killing and reducing their attachment. These materials can be applied to antiviral textiles through finishing and various spinning methods. In this paper will experimentally study the efficiency of sulphate-based compound as antimicrobial, fluorinated-based compound as water-repellent, synthesis and efficiency of Graphene nano-composite as anti-bacterial &antiviral treatment, also will study the method and conditions to bind the listed treatment substances to the textile fabrics.

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Keywords: Medical protective clothing, cotton fabric, antibacterial, antiviral

Introduction

In terms of emerging diseases control, biological protection textiles play an important role. Reports have shown that they can weaken the spread of viruses and reduce the sudden dangerous occurrence of the pandemic virus through the utilization of protective clothing, medical-surgical masks, and N95 masks. However, although protective clothing and masks can reduce the spread of the virus, the risk of infection cannot be eliminated. Antiviral textiles can kill viruses on the surface of the fabric or inhibit the formation of biofilms, reducing the risk of infection/re-infection. Moreover, antiviral textiles can reduce environmental pollution due to their reusability. However, most textiles with antiviral properties have not been industrialized, and there is no systematic research on antiviral textiles. Therefore, the development of antiviral textiles is urgent.

Cotton is one of the most widely used fabrics in the manufacture of Medical protective clothing which Sterilizable and reusable.

It is known that untreated cotton fabrics are a favorite target for bacteria attack, so the aim of this work was to add new properties that increase the value of cotton cloth, especially if the materials that will be added to the cloth do not need external preparation, but rather are prepared directly on its surface. The cotton fabrics were treated with graphene nanoparticles.

And since the main objective of the research is to enhance the performance of medical protective clothing with the following things:

• Developing a suitable fabric of cellulose materials (100% cotton) for surgical gowns.

• Apply a nano-coating to the surface of the fabric.

• Transfer of Anti-bacterial and anti-viral property to the tissue.

• Evaluation of the Anti-bacterial and anti-viral properties of the coated tissue.

This is as follows:

Chemicals & materials

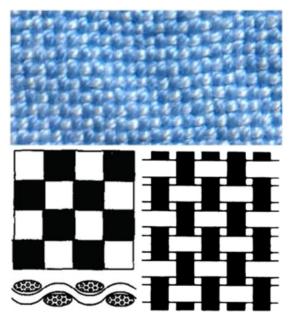
PU emulsion polymer "SPI Company." Anti-bacterial, sulphate compounds "BASF

Company".

Graphite rods "SPI Company."

Fluorinated compound "BASF Company".

White textile fabric "100% bleaching cotton fabric (Giza 86, Plain weave 1/1, 144 gm/m 2, Number of threads in the warp 84, Number of threads in the weft 66, count number yarn of warp 24/1, count number yarn of weft 24/1).



Caption (1): Histological structure of a cotton sample $(plain \ fabric)^1$

Tools and equipment's

General laboratory equipment's Magnetic stirrer Power supply 7volts Convert-belt dryer Padding unite Ultra sound miller

Methodology

The fabric was washed and dried to be suitable for treatment, then treated with the pre-synthesized liquor using manual padding unit at room temperature, the fabric then squeezed, dried and fixed at certain conditions according to each treatment, all samples were measured, the results were reported in tables and clarified with figures.

Procedures

Studying the efficiency of anti-bacterial treatment

Four samples were treated with anti-bacterial compound with concentrations of 0.25%, 0.5%, 1% and 1.5%, as mentioned in methodology, the samples were dried and fixed at $80C^{\circ}$ and $150C^{\circ}$ respectively, the results were reported in table (1) and clarified with figure no1.

Test method:

The test was done by Gram Stain Test, one of the most important types of dyes used in hospitals to identify the type of bacteria. Thanks for its discovery to the doctor of Danish origin Hans Christian Gram (Hans Christian Gram). Where he developed this method to help him differentiate between the types of bacteria that cause pneumonia, where he noticed that one type of bacteria dyed red when placed in a specific solution containing iodine and called it (gram-negative bacteria), and another type of bacteria was blue in color It was called Gram-positive bacteria. The color of bacteria in a Gram stain depends on the chemical composition of the cell wall.²

The steps for staining bacteria with a Gram stain include: $^{\rm 3}$

Get a glass test slide and swab it for bacteria.

Stabilizing the bacteria on the slide, by passing the slide over the flame several times.

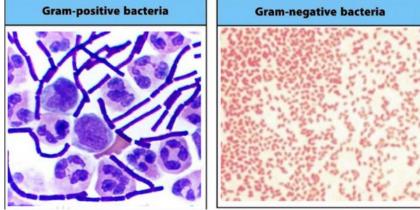
Placing the basic dye, Crystal Violet, on the slide for one minute, and the bacteria will acquire a violet color.

Wash the slide with water; To remove excess dye from the slide.

Then the slide was rinsed with iodine solution; To fix the dye, followed by a solvent

Different bacteria react differently to the staining method. So, bacteria are divided into:

- Gram-positive: It is generally dark blue
- Gram-negative: remains a gram without coloring but is colored red by fuchsin



Caption (2): Gram-positive and Gram-negative bacteria⁴

Studying the efficiency of water-repellent treatment

Four samples were treated with fluorinated composite with the

Concentration of 120,130,140 and 150 gm./L, as mentioned in methodology, all samples were dried and fixed at $80C^{\circ}$ and $150C^{\circ}$ respectively, the results were reported in table (2) and clarified with figure no2.

AATCC 22 and UNE EN ISO 4920: Water repellency – Spray test

The water repellency test was carried out by a "spray test" in accordance with ISO 4920 / EN 24920.

Testing is carried out by the following components:

- 19-hole spray gun

A glass funnel of a certain diameter

- Circular support inclined at an angle of 45 degrees for testing on test tubes;

The distance between the center of the atomizer and the center of the support is fixed as specified in the specification.⁵



Caption (3): Shape of wetness meter ⁶

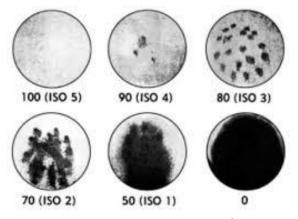
The test is carried out by fixing the fabric to the frame so that it does not fold, then pouring 250 ml of water at (20 ± 5) °C into the funnel; so, you will have an average spray length of 25-30 seconds. Then he rotates the tire 180 degrees and the test is repeated.

Principle: The resistance of the cloth to surface wetting is the extent of the wettability of the surface as a result of exposing it to a spray of water, and it is expressed by a certain degree of wetness or by the percentage of its weight gain, and therefore a specific volume of distilled water is sprayed on the test sample that is installed above a metal ring tilted at an angle of 45 ° so that the center of the sample is on Specific distance from the sprinkler nozzle, the

degree of wetness is determined by comparing the appearance of the sample with standard descriptive samples and photographs.

Procedures:

At least 3 samples are prepared with dimensions (200 x 200 mm) to be taken from different places of the fabric. We fix each sample on the upper face of a frame, then put the frame on a support so that the length of the sample is parallel to the direction of water flow and fall into the sample. The funnel is filled at once with an amount of (100) ml of distilled water, then the sprayer is stopped directly, and the sample is taken with the catcher and hit twice lightly on a solid object to shake off the sample. We determine the percentage of wetness of the sample by comparing it with the following descriptive measurement:⁷



Caption (4): Standard Spray Test Ratings⁸

Test results:

Based on the AATCC normative website.

1- (100) ISO 5: There is no wetting on the surface of the fabric.

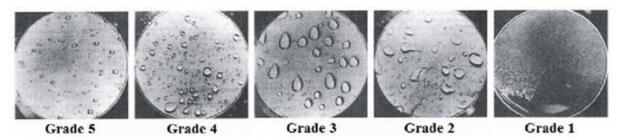
2- (90) 4 ISO: There is a random and light wetting on the surface of the fabric.

3- (80) 3 ISO: There is a wetting of the surface of the tissue in the form of points (point).

4- (70) 2 ISO: There is one primary fraction on the entire surface of the tissue.

5- (50) 1 ISO: There is complete wetting on the surface of the (upper) fabric.

6- (0) 0 ISO: There is complete wetting on the surface of the tissue (upper and lower).⁹



Caption (5): standards used for assessing the water repellency of fabrics by the Bundesmann rain-shower test¹⁰

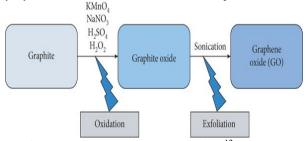
Studying the efficiency of anti-bacterial & water-repellent composite "one component treatment.

Four samples were treated with a pre-synthesized mixture of 0.5% anti-bacterial and different concentration of water-repellent substance "120,130,140 and 150 gm./L", the samples were dried and fixed at 80° and 150° respectively, and all results were reported in table (3) and clarified with figure no3.

Synthesis of grapheme nano-particles Synthesis of GO

- 1. Brodie-Staudenmaier-Hummers Based Methods
- 2. Tour Method
- 3. Free-Water Oxidation Method
- 4. Monolithic Crystalline Swelling of GO

To sum up, there is no specific method or procedure for producing a "standard" GO because each synthesis method produces a different GO type. Therefore, the GO has distinct physicochemical properties, such as structure and reactivity.¹¹



Caption (6): Diagram of GO preparation.¹²

Graphene was synthesized using electro chemistry technique using graphite and platinum rods; both graphite and platinum were connected with power supply 7 volts, the graphite rod start the to decay into the ionized aqueous solution, the synthesized liquor was filtered using vacuum filtration and washed out with distilled water 3 times, the obtained powder was subjected to reduction process for 30 min. at 400C°. The synthesized graphene nano- particles was measured to ensure about the size and molecular distribution, the results were clarified with chart no.1 and figure no. 4

Studying the anti-viral efficiency of graphene nano-composite

Various emerging infectious diseases caused by viruses, including severe acute respiratory syndrome coronavirus (SARS-CoV), Ebola virus, norovirus, and dengue virus have prompted the discovery and development of antimicrobial reagents and personal protection equipment (PPE) to guard against infectious agents. Silver nanoparticles (Ag NPs) have been proven to be the most effective antimicrobial agents against bacteria and viruses because of their high surface-area-to-volume ratio and unique chemical and physical properties, even though they have shown cytotoxicity at high concentrations.^{13,14}

Graphene nano-particles was mixed with PU as a fixing agent and non-ionic dispersing agent, and then subjected for 4 hours to ultra sound milling process.

The tested fabrics were treated with the synthesized Graphene liquor by padding technique, squeezed and finally subjected to heat fixation at 130Co for 2min.

Different graphene concentrations 25, 50 and 75 were applied.

The tested samples were measured; the anti-viral results were reported in table (4) and clarified with figure no. 5

Results & discussions

The results of Water repellent& soil release, anti-bacterial and anti-viral treatments were studied and discussed to ensure about the efficiency of each one, the experiments were done with different concentrations of each material as follow

1. Studying the effective concentration of anti-bacterial treatment.

Recommendation	S. aureus		E. coli	
	Colonies No.	Reduction	Colonies No.	Reduction
Sample No	<i>CFU x10⁵</i>	%	$CFU x 10^7$	%
Blank	5.7	0	26.3	0
%0.25	1.104	80.63158	12.09	54.03042
%0.5	0.87	84.73684	13.01	50.53232
%1	0.61	89.29825	8.7	66.92015
%1.5	0.092	98.38596	1.992	92.42586

Table (1).Relation between anti-bacterial conc. and reduction efficiency

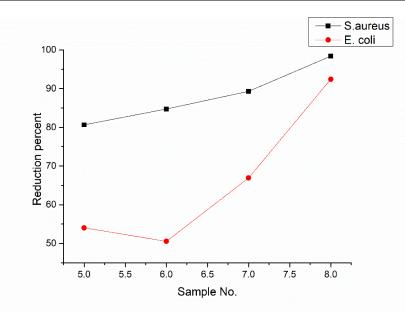


Figure (1): .Relation between anti-bacterial conc. and reduction efficiency

From the illustrated table (1) and figure (1) found that: the reduction of bacterial growth increased with the increase of the anti-bacterial compound concentration; generally, all recipes starting with 0.5% achieving an acceptable result, the **2. Studing the efficiency of water repellent& soil release treatment**

The water-repellent property is one of the important properties, as it expresses the ability of the fabric to resist water and various liquids that doctors and medical workers may be exposed to.

Table (2) and figure (2) show the test results. By discussing these results, it was found that the sample with concentration came in the first place, as it gave a reading of 0° (i.e., total wetness of the surface and bottom of the material), and samples No. (3) and No. (7) came in the second place, as it gave a reading of 50° (i.e., total wetness of the surface). Then came the third and final samples No. (1), No. (4), No. (5), and

maximum reduction achieved at 1.5, while at 0.5% concentration the reduction of E. coli showing un asymmetrical point, it may be due to mistake in sample preparation, will ensure about this point or forget it.

No. (6), as each of them gave a reading of 70° (i.e., partial wetness of the surface).

This is due to the opening of the tissue structure (rib) of sample No. (2), as well as to the material in this sample, which is micro modal, which makes the brown spaces between the hairs within the thread itself very large, allowing it to absorb water and expel it to the other side of the fabric easily.

Finally, the illustrated table and figure showing that: all concentrations of the treatment composite achieve an acceptable result as the concentration of 130gm/l achieves the maximum water repellent result, so no need to apply such higher concentration.

Table (2):.Studying the efficiency of water-repellent treatment

Samples	Blank	120 gm\L	130gm\L	140 gm\l	150 gm\L
Results	0	90	100	100	100

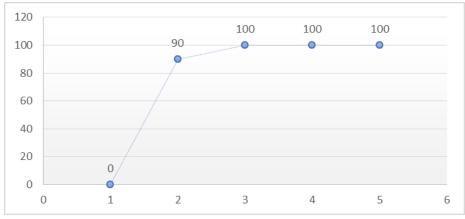


Figure (2) .Studying the efficiency of water-repellent treatment

3. Studying the efficiency of anti-bacterial & water-repellent composite "one component treatment.

Table (3) and figure (3) showing that: at 0.5% anti-bacterial with different ratios of water-repellent composite, the reduction of bacterial growth increased with the increase of the concentration of water-repellent compound, meaning that the water-repellent has a positive effect, so the use of both components together as one-component is recommended.

4. Studying the particle size of synthesized graphene

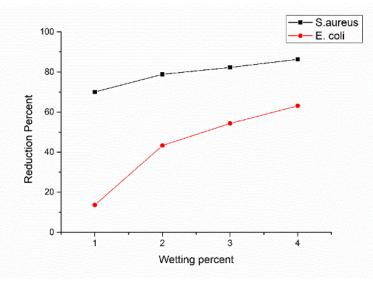
The synthesized graphene particles were measured to ensure about the exact size and also the molecular size distribution, the obtained results were as follow:

Zeta Potential Analysis

The prepared graphene nanoparticles solutions were characterized for their polydispersity and zeta potential, the obtained results are presented in Table (4) and clarified with illustrated charts and photos.

Recommendation	S. aure		E. coli		
Sample	Colonies No. Reduction		Colonies No.	Reduction	
No	CFU x10 ⁵	%	CFU x10 ⁷	%	
Blank	5.7	0	26.3	0	
120gm\L	1.706	70.07018	22.7	13.68821	
gm\L130	1.206	78.84211	14.9	43.34601	
gm \L140	1.01	82.2807	12.01	54.3346	
Gm\L150	0.78	86.31579	9.7	63.11787	

Table 3. Studying the efficiency of 0.5 % anti-bacterial at different conc. of water-repellent composites.



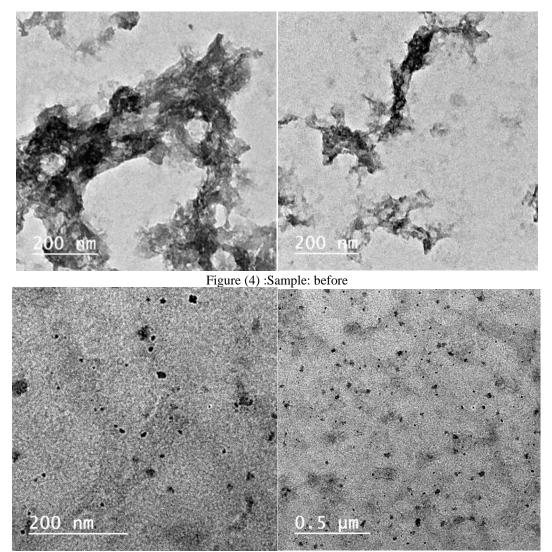


Figure (3): Studying the efficiency of 0.5 % anti-bacterial at different conc. of water-repellent composites.

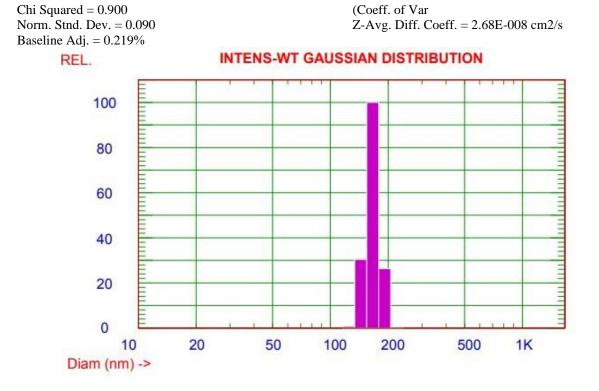
Figure (5): Sample: after

-	Samples	Polydispersity index	Zeta potential (mV)
	Before	0.247	-1.99
	After	0.095	-13.37

The results show that the polydispersity index showing homogenous particle size distribution of the prepared graphene particles within the solution. The Zeta potential values varied in the range of -1.99 mV before to -13.37 mV. Zeta potential value in sample before was very low showing that the solution may be doesn't have high stability as nanoparticles and high probably it starts to aggregate. Sample after showed the highest zeta potential value and it can be indicated that it is the most stable of all tackled solutions.

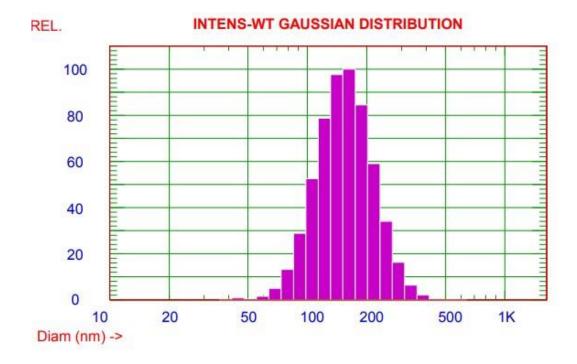
Sample: Before

INTENSITY-Weighted GAUSSIAN DISTRIBUTION Analysis (Solid Particle) Mean Diameter = 166.4 nm Variance (P.I.) = 0.008 Stnd. Deviation = 15.0 nm (9.0 %)



Cumulative Result :

% 25 of distribution < 155.9 nm % 50 of distribution < 165.7 nm % 75 of distribution < 176.0 nm % 90 of distribution < 185.9 nm % 99 of distribution < 204.2 nm % 80 of distribution < 178.7 nm Run Time = 0 Hr 1 Min 10 Sec Wavelength = 632.8 nmCount Rate = 430 KHzTemperature = 30 degChannel #1 = 184.7 K Viscosity = 0.995 cp Channel Width = 19.0uSec Index of Ref. = 1.333Sample: After Mean Diameter = 162.8 nmVariance (P.I.) = 0.108Stnd. Deviation = 53.6 nm (32.9%) Chi Squared = 1.201Norm. Stnd. Dev. = 0.32Baseline Adj. = 0.000 %(Coeff. of Var'n) Z-Avg. Diff. Coeff. = 2.74E-008 cm2/s



Cumulative Result:

% 25 of distribution < 123.5 nm % 50 of distribution < 154.2 nm % 75 of distribution < 192.5 nm % 90 of distribution < 235.0 nm % 99 of distribution < 331.5 nm 80 % of distribution < 203.4 nm Run Time = 0 Hr 1 Min 10 Sec Wavelength = 632.8 nm Count Rate = 306 K Temperature = $30 \deg C$ Channel #1 = 92.0Viscosity = 0.995 cp Channel Width = 19.0uSec Index of Ref. = 1.333

5. Studying the anti-viral efficiency of graphene nano-composite

Three concentrations of graphene composite were applied into the test fabrics; the efficiency results of each concentration were as follow:

Conc.	of	Duration	of	Virus	Control	Vira	l Titer	Viral Inhibition (%)
Graphene	on	incubation	with	withou	t treatment	Post-Trea	tment	
tissue pe	r mg		virus		(PFU/ml)	(PF	U/ml)	
	25		20 min		$3.0 * 10^{6}$	1.4	* 106	53.3 %
	50			1.0 * 10	6	2.2	* 105	78 %
	75			1.0 * 10	6	1.0	* 10 ⁵	90 %

Table 5. The effectiv	a concentration o	f anomhana aom	monito on optiving
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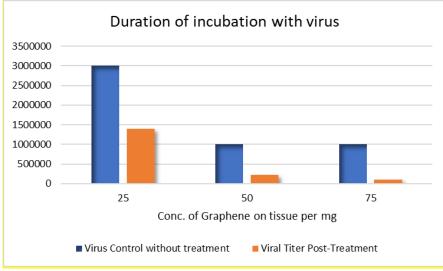


Figure (6): Relation between graphene concentration and virus reduction

The treated tissue samples with graphene showed that high antiviral activity against Severe Acute Respiratory Syndrome Coronavirus 2 (SARS-CoV-2) with inhibition percent 90% at concentration 75 mg after incubation period 20 min, 78% with 50mg and 53.3% with 25 mg post-treatment for 20min.

Anti-Viral activity according to ASTM E1053-20:

The virus Severe Acute Respiratory Syndrome Coronavirus 2 (SARS-CoV-2) hCoV-19/Egypt/NRC-03/2020 (Accession Number on GSAID: EPI ISL 430820) virus suspension is applied on an inanimate, nonporous surface. The test substance is added over the film. Control carriers receive the same condition. After exposure at the appropriate temperature (usually 20 ± 2 °C) for the recommended time, the elutes from control and test carriers are assaved for infectivity by plaque reduction assay.

From the antiviral study found that all the concentrations have a positive effect against Coronavirus 2 (SARS-CoV-2), while the concentration of 75 achieves the best inhibition result.

Plaque reduction assay

Assay was carried out according to the method of (Hayden et al., 1980). In a six well plate where Vero E6 cells were cultivated for 24 h at 37°C. The control untreated and treated virus was incubated with the tested sample for 20 min and 3 different concentrations (incubation sample with virus for:20 minutes with concentrations 25,50 and 75 from **graphene**). Growth medium was removed from the cell culture plates and the cells were inoculated with (100 μ / well) virus dilution. After 1 h contact time for virus adsorption, 3 ml of

DMEM supplemented with 2% agarose was added onto the cell monolayer; plates were left to solidify and incubated at 37°C till formation of viral plaques (3 to 4 days). Formalin (10%) was added for two hours then plates were stained with 0.1 %crystal violet in distilled water. Control wells were included where untreated virus was incubated with Vero E6 cells and finally plaques were counted and percentage reduction in plaques formation in comparison to control wells was recorded as following:

% inhibition= viral count (untreated) - viral count (treated)/viral count (untreated) x 100

Conclusion:

The treated tissue samples with graphene showed that high antiviral activity against Severe Acute Respiratory Syndrome Coronavirus 2 (SARS-CoV-2) with inhibition percent 90% at concentration 75 mg after incubation period 20 min, 78% with 50mg and 53.3% with 25 mg post-treatment for 20min.

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