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The Effect of Different Nanoparticles on Dyed Wool Carpet with Walnut Shell Natural Dye

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ABSTRACT

Nowadays, research on handmade carpet is developing. It is very important to protect dyed fibers by natural dyes in handwoven woolen carpets against harmful environmental bacteria. In this study, the effect of different nanoparticles on dyed woolen carpet has been investigated. Wool yarns were mordanted with alum 10 % and acetic acid 2 % and then dyed with 60 % walnut natural dyes. After dyeing, carpet was woven and finally was treatment in three steps with three types of particles (nano-silver, nano-titanium dioxide and nano-alumina oxide). SEM, FTIR, color value and color strength, self-cleaning, moisture absorption time, the size of the contact angle, anti-odor property and anti-bacterial activities of dyed wool carpet are investigated. The results showed that the hydrophilic properties of samples have been improved. SEM images indicated the presence of nanoparticles on samples. Also, antibacterial properties of samples against E. coli and S. aureus were very good evaluated. Moreover, Long-term static loading was applied on the sample to make the pile up before and after loading. Prog. Color Colorants Coat. 16 (2023), 231-241© Institute for Color Science and Technology.

1. Introduction

J. regia (common walnut) is a deciduous tree of the Junglan-daceae family native to south-eastern Europe. It reaches a height of up to 25−35 m and a diameter of up to 2 m. The water extracted from walnut leaves is reported to exhibit powerful antioxidative and antimicrobial properties owing to the presence of large amounts of phenolic compounds, such as naphtoquinones and flavonoids. From among the naphtoquinones, the juglone is of great importance due to its chemical reactivity [1].

The chief coloring compound present in walnut dye is juglone having molecular formula $C_{10}H_6O_3$. Chemically the molecule of juglone is 5-hydroxy-1, 4-naphthoquinone and is described as natural brown (juglone, CI 75500), which acts as a substantive dye and imparts brown color to the textile substrates. Juglone is a quinone group aromatic compound has been found in all parts of the walnut tree and it has also been the amounts of juglone were in order of green peel>leaves>bark and it has phytotoxic and allelopathic properties [2-3]. Figure 1 show the main chemical structure of walnut natural dye.

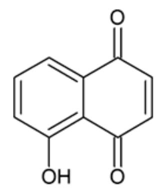

Figure 1: Chemical structure of juglan in a walnut hull [4].

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Natural dyes are highly regarded for their chemical composition due to their lack of chemical hazard and toxic effects. Therefore, for many years, the use of renewable and environmentally friendly resources has been considered [5]. In recent years, studies have been conducted on the antifungal activity of phenolic compounds including flavones and related flavonoid glycosides, coumarins and derivatives, and anthraquinones. Presence for a large number of tannins in some of the common plant derived nature dyes and naphthoquinones such as lawsone in henna, juglone in walnut and lapachol in alkannet are reported to exhibit antibacterial and antifungal activity. The antifungal potency of natural dye derived from B. prionitis aerial parts may be due to the presence of phenolic chemical constituents [6].

It is known that naphthoquinones are slightly soluble in water. A small amount of juglone (a quinone form) dissolves in water. When juglone is subjected to aqueous NaOH solution, hydrogen is replaced by metal in the hydroxyl group in the aromatic ring (a phenolate form). A phenolate form of juglone becomes water-soluble [7].

Natural dyes having functional groups such as −OH, $-NH_2$, −COOH, etc. groups and the textile fibers with active sites (–OH, $-SO_3H$, −COOH, $-C_6H_5OH$, $-NH_2$) can make a complex with or without mordant. Because of the different interactions of dye and fabric, many functional properties can be achieved. Besides functional group of natural dye such as tannins, flavonoids, anthraquinone, etc., functional groups of respective fabrics are also responsible for functional properties in the fabric [8].

An interest in the use of natural dyes in textile coloration has been rising and this is a result of the rigid environmental standards imposed by many countries in a response to the toxic and allergic reactions associated with synthetic dyes [9]. In this regard, some researchers improved application of natural dyes along with nanoparticles to finding definite properties on wool samples [10].

In this study, we investigated the effect of different nanoparticles on dyed wool with walnut natural dye. Then, morphological properties, color characteristics, self-cleaning, antibacterial activities and some other properties were evaluated.

2. Experimental

2.1. Material and equipment

In this study, merino wool yarn with a score of ten folds, walnut natural dye, nonionic soap (Nikogen SDN), deionized water, mordant alum, acetic acid, hypophosphite and sodium carbonate from Merck (Germany) were used. Also, nano silver with an average particle size of 20 nm, nano dioxide Titanium with a purity of 90 % and a particle size of 20 nm, and nano aluminum oxide with a purity of 99 % and a particle size of 50 nm, were obtained from US Research nanomaterials. X-rite sp62 reflective spectrophotometer, Phenom Prox SEM scanning electron microscope, Spectron 700 transfer spectrophotometer, Camera Video Color- spectrometer series Made in USA Nexus-Spattering, and Colony counter were used for additional analyses.

2.2. Method

Merino wool yarns were first washed in a bath containing 2 g/L nonionic soap at 50 °C for 20 minutes with a L:R=40:1. For mordanting, samples with the alum mordant 10 % and acetic acid 2 % were treated at 100 °C for 45 minutes (Figure 2). Then, the mordanted samples, dyed with the natural dye (60 % o.w.f) at 100 °C for 1 hr (Figure 3).

Finally, after dyeing step, the samples in solution containing 2 g/L nonionic soap at 50 °C for 10 minutes were washed. After washing step, the samples were woven 50 centimeters in 50 centimeters with 50 rings. Woven samples treated at three stages with various nanoparticles.

In the first stage, the woven samples were placed inside a nano silver solution with 0.5 and 2 g/L and acetic acid 10 % (20 % o.w.f) at 80 ºC for 15 minutes, then samples was rinsed with deionized water and cured at 100 °C for 10 minutes. In the second step, the samples treated in the solution containing acetic acid 5 % (10 % o.w.f), Sodium hypophosphate 2 % (6 % o.w.f) and nano dioxide titanium as 0.5 and 1.5 g/L at 90 °C for 30 minutes. Treated samples was rinsed with deionized water, and then were cured for 2 minutes at 120 °C.

Finally, Samples were placed in sodium carbonate 3 % (5 % o.w.f) and nano oxide aluminium as 0.5 and 1.5 g/L at 60 ºC for 15 minutes and then washed with deionized water and cured in an oven at 100 °C for 10 minutes. Table 1 indicate the condition of treated samples in the various stages.

Figure 4 indicate the mechanism of interaction between wool, mordant and dye molecule. The oxygen in a polar group of natural dye has a strong affinity for silver ions and silver nanoparticles. The last stage (after coating of dyed samples with all nanoparticles) can involves the formation of coordinative bonding between the oxygen of a hydroxyl group in juglone dye and nanoparticles.

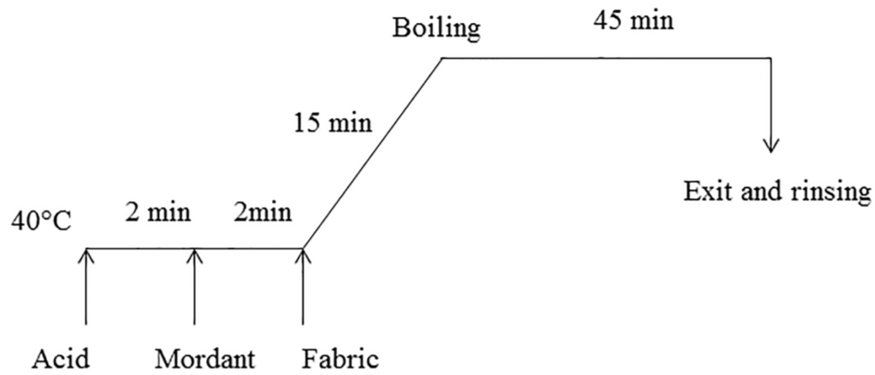

Figure 2: Mordanting diagram.

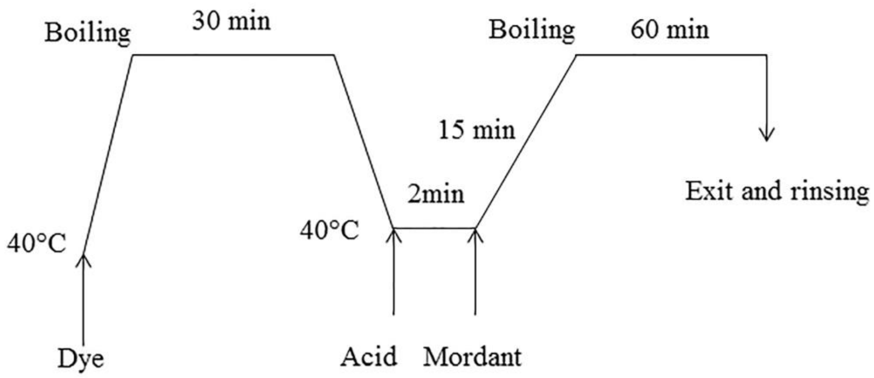

Figure 3: Dyeing diagram.

Table 1: Code of samples and quantities of used materials Walnut shell dye (60 % o.w.f).

Material	G1	G2	G3	G4	G5	G6	G7	G8	G9
Alum mordant %	-	10							
Acetic acid %	-	2							
Nano silver (g/L)	-	2	2	2	2	0.5	0.5	0.5	0.5
Acetic acid 10 %		20 %							
Nano dioxide titanium (g/L)	-	1.5	1.5	0.5	0.5	1.5	1.5	0.5	0.5
Acetic acid 5 %	-	10 %							
Hypophosphite sodium 2 %	-	6 %							
Nano oxide aluminium (g/L)	1.5	0.5	1.5	0.5	1.5	0.5	1.5	0.5	1.5
Carbonate sodium 3 %	-	5 %							

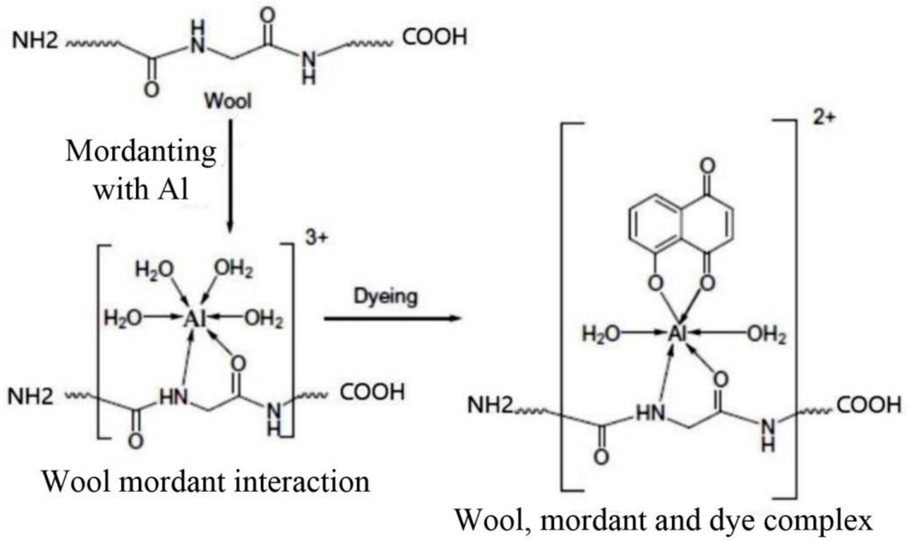

Figure 4: Complex formation between wool, mordant and dye molecule [2].

3. Results and Discussion

3.1. Investigation of color values, self-cleaning properties and color strength

Application of natural dyes on fabric significantly enhances the UPF of the fabric. UPF of fabric is affected by the absorption characteristics of natural dyes. Tannins in plant act as a chemical protector against the UV radiation. Tannin as a phenolic compound absorbs UV radiation due to resonance in structure and hence after the process of attachment on textile surface. These attached molecules absorb the UV radiation and thus protect the skin from absorbing UV rays. The presence of hydroxyl and carbonyl groups in tannins is responsible for forming a large number of hydrogen bonds with fabric and also showed van der Waals forces with fabric, thus improving the color strength of fabric [11]. Tannin-based natural dyes such as R. maritimus, M. philippinensis, K. lacca, A. catechu and A. nilotica have good UV protection properties Ferrous sulphate as a mordant establishes ternary complex with both fiber and the dye, and the remaining coordination sites of Fe metal can absorb UV radiation by converting electronic excitation energy into thermal energy [8].

The color components of the samples were measured with the ASTM D 1925 standard by x-rite sp 62 reflectance spectrophotometer in the range of 400-700 nm with a standard viewing angle of 10 degrees and a D65 light source. All samples were exposed to ultraviolet light for 48 hours after finishing with nanoparticles to evaluate self-cleaning properties. Color strength (K/S) of samples was calculated based on the Kubelka-Munk relation.

Ali Khan et al. [2] confirmed that the walnut dye extract can give satisfactory results in wool dyeing, even without mordanting; also, the use of potassium aluminum sulphate as a mordant in wool dyeing with walnut dye extract resulted in an improvement in the K/S and fastness properties of dyed wool.

Color difference and self-cleaning were investigated using the following equation (CIE L*a*b* 1976) (Eq. 1).

$$\Delta E=\sqrt{\left(\Delta L^{*}\right)^{2}+\left(\Delta a^{*}\right)^{2}+\left(\Delta b^{*}\right)^{2}} \quad (1)$$

In Table 2, the values of the color components and the color strength before and after exposure is stated. In most cases with an increase the amount of silver, titanium dioxide and oxide aluminum nanoparticles increase the brightness of the samples. The amount of redness and yellowness in most samples decrease and, in some specimens, after exposure the ultraviolet light has not changed. Comparing the brightness of the control sample with other samples, the results show that in the G6 sample the maximum brightness is obtained. In other words, rising of L*, indicates an increase in fading due to the self-cleaning property is the result of the presence of nanoparticles.

Table 2: Values of color components, color difference and color strength of treated samples before and after exposure.

code	L*	a*	b*	K/S	R	code	L*	a*	b*	K/S	R
G1	32.22	9.65	16.39	20.33	2.49	UVG1	38.05	10.68	18.38	13.01	3.75
G2	36.3	7.05	11.09	8.12	5.83	UVG2	42.55	7.27	11.17	6.44	7.27
G3	38.2	8.28	13.17	9.91	4.82	UVG3	39.7	8.5	13.18	9.11	8.28
G4	35	7.9	13.03	12.65	3.82	UVG4	38.49	7.64	12.85	10.36	4.69
G5	36.26	8.08	13.07	11.4	4.26	UVG5	38.47	8.35	14.02	8.12	5.88
G6	39.84	8.8	13.62	8.42	5.61	UVG6	41.73	8.77	13.06	7.07	6.64
G7	35.84	7.15	11.72	6.62	4.31	UVG7	38.59	7.32	12.79	8.12	5.81
G8	36.32	7.46	12.11	10.61	4.58	UVG8	40.81	7.56	12.31	7.97	5.93
G9	36.55	9.75	15.18	12.65	3.87	UVG9	36.02	8.82	14.59	10.8	4.45

As can be shown that in Table 2, with increasing the amount of titanium oxide nanoparticles and decreasing the concentration of aluminum oxide nanoparticles, the color difference of the samples and the self-cleaning properties of the carpets have increased. In other words, titanium dioxide nanoparticles have higher self-cleaning properties (more severe oxidizing properties) which is due to the fact that when the nanoparticle optical catalyst coating is irradiated with light, hydroxyl and super-oxidizing radicals produced on the surface are compounded. And cause self-cleaning properties [10].

3.2. Investigation of contact angle and water droplet adsorption time

Surface wettability is one of the most important characteristics of surface quality. The most important characteristic for measuring this feature is the contact angle. Surface roughness is also the most important structural feature of the surface that causes changes in surface wettability. Surface energy is also largely dependent on the surface material. Therefore, changing the surface roughness can be the main goal in achieving surfaces with special hydrophobic and hydrophilic conditions. The hydrophilicity of titanium oxide nanoparticles causes water droplets to always be placed at very low angles on surfaces coated with titanium oxide nanoparticles, tending to spread on the surface. 10 The hydrophilic nature of titanium oxide nanoparticles allows water droplets on surfaces coated with titanium oxide to be always at very low angles and tend to spread on the surface and get wet.

The duration of water droplet adsorption was measured by the standard method AATCC Test Method 79-2000 and to reduce the amount of test error, was performed at four different points of the carpet and the angle of contact of water with the samples was measured with Video Camera Color. Figure 5 shows the contact angle of treated samples. Absorption and desorption of water from wool with the number and type of functional groups in chains protein is associated. On the other hand, the hydrophilicity of nanoparticles depends on their specific surface, the average particle size and how to place the particles in gatherings.

The lowest contact angle and water droplet adsorption time is related to the sample treated with the highest concentration of all nanoparticles (G2 sample). As can be seen from Figure 6, the treated sample with the lowest value of all nanoparticles (G9 sample) attained the highest adsorption time.

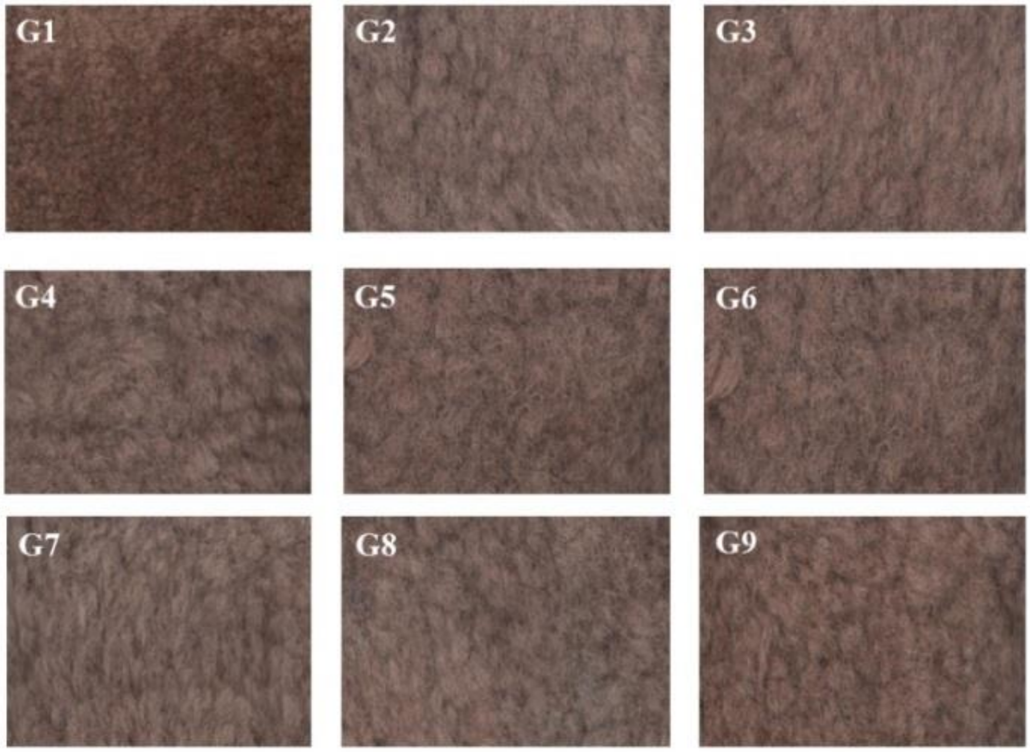

Figure 5: Scanned images of woven carpet.

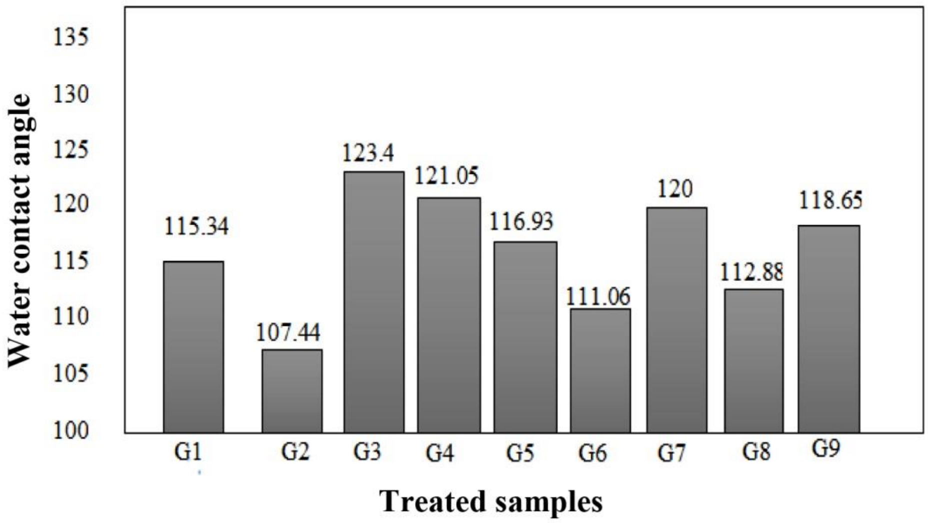

Figure 6: Water absorption time of treated samples.

3.3. Evaluation of detoxification

Natural dyeing of cotton, wool and silk fabric using gallnut also provides a better deodorizing function against ammonia, trimethylamine and acetaldehyde and showed bacterial resistance against Staphylococcus aureus and Klebsiella pneumonia. The main component in the gallnut extract was found to be Gallotannin which is the reason for these functional properties [8].

Human olfactory test was performed according to ISO standard at 22±3 °C and relative humidity R.H= 7%±38 and processed samples with 200 grams of chopped onions are kept in a sealed container for 24 hours [12]. After 24 hours, the samples were removed from the container and each one was placed inside the human with a lid. In this case, the samples were then given 1 hour to balance their upper space for human olfactory testing [13].

The samples were evaluated by 9 people and the obtained numbers are the average of this evaluation. These numbers show that due to the use of aluminum nanoxide in the final stage of completion, the samples have been significantly deodorized. (G5, G6 and G7 samples indicate the highest odor intensity and also G1 and G2 samples indicates the lowest odor intensity based on human sense of smell (Figure 7).

3.4. Investigation of surface morphology of samples using scanning electron microscopy

SEM were performed on four samples (raw wool, walnut dyed sample (G1), walnut dyed sample and supplemented with the highest (G2) and lowest (G9) nanoparticles (Figure 8).

Figure 9 displays that raw wool (untreated wool) and G1 sample have perfectly defined scales with clear edge. Moreover, existence of nanoparticles and their submicron aggregates in surfaces of finished samples (G2 and G9) with uniform distribution and small particle size about 66 nm can be seen.

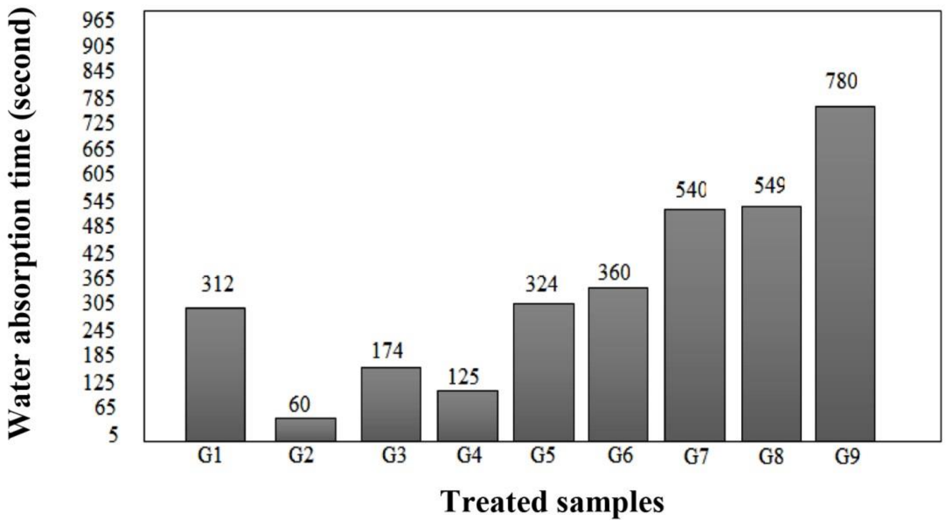

Figure 7: Water absorption time of treated samples.

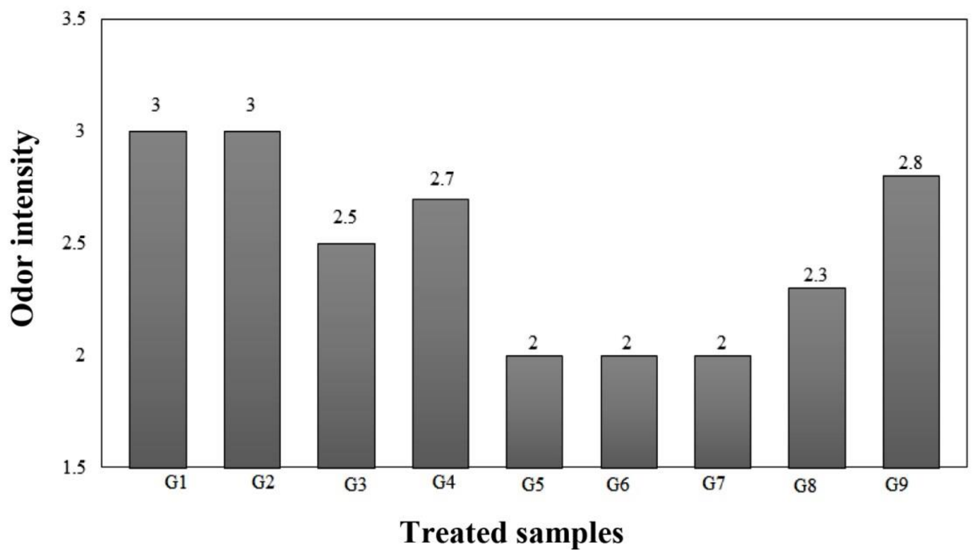

Figure 8: The odor intensity treated samples.

3.5. Static loading

Carpets are usually exposed to two types of loads during use: static (such as table and chair bases) and dynamic (such as walking on the carpet, moving furniture on the carpet). Carpet yarns are compacted in this constant or fast compressive load, their deformation is complex, and each villus are individually subjected to different deformation modes due to axial pressure, bending, flattening, stretching and shearing. Compressive behavior of carpet lint during loading and reaction. Their dynamics after load removal have an important role in their mechanical performance. Static loading machine with force arm has been used on the same basis from 1966 until today. Often in this machine, a horizontal arm with an end weight is designed to apply a load with a length ratio of 1: 5 to the arm in contact with the product. five times the amount of weight added to the end of the horizontal arm will apply a constant compressive load to the sample.

Standing and walking are different forces and deformations that applied to carpet piles during such human daily activities. Axial compression, bending, flattening, and extension and also static and dynamic pressure are some examples of these deformations. Furthermore, the compression behavior and mechanical reaction influence on the piles thickness loss in the carpet. The recovery behavior of the pile carpet after static loading is important for the quality, performance and lifetime of the piled carpet after static loading [10].

Carpet is predominantly used in home floor covering as an indispensable decorative product and also preferred by its heat and sound insulation feature. Compression performance in general terms of mechanical properties influences carpet performance under dynamic or static loads. Carpet thickness will be deformed when it is exposed to dynamic and static loads which are created by walking and furniture, respectively. During daily usage of the carpet, thickness loss is directly affected by raw material, pile height, carpet construction, pile density etc. There are a lot of studies focused on effects of these parameters on carpet performance based on static loading, dynamic loading and compressibility [14].

This experiment was performed by long-term loading method with a compressive load period of 24 hours. This test is in accordance with BS ISO 10834 standard. The sample (G2) was subjected to a compressive load of 700 kPa for 24 hours by a static loading device. After loading, the villi height of the sample was measured and recorded. Result in Table 3 indicate that after unloading, the villi height of the sample decrease.

3.6. Investigation of antibacterial properties

The surrounding of human includes a variety of microorganisms like bacteria and other microbes, which are invisible for the naked eyes. Bacterium is a unicellular organism. It is divided in Gram-positive like Staphylococcus aureus (S. aureus) and Gram-negative like Escherichia coli (E. coli) on the basis of chemical and physical properties of their cell walls. It is reported that antibacterial and antifungal properties of natural dye are due to its phenolic content. Phenolic compounds attach on the surface of textiles by forming a complex. When the fabric comes in contact with microbes, these attached phenolic compounds hamper the enzyme production in microbes; thus, further cell reaction would not take place and at the end cell dies.

Rumex maritimus (golden dock) and Quercus infectoria (indigo) dyes are found to be the most effective against common microbes like E. coli, Bacillus subtilis, Klebsiella pneumoniae, Proteus vulgaris and Pseudomonas aeruginosa. Berberine compound as a cationic dye having quaternary ammonium structure can act as an antibacterial agent. The dyed wool represented a high level of antibacterial activity. Application of natural dyes like pomegranate, clove, coptis chinensis (Chinese goldthread) and gallnut extracts on cotton, silk and wool fabric provides excellent antibacterial activity against S. aureus.

Table 3: Height of carpet sample lint.

The height of the villi, 1 minute after unloading (cm)	Initial height of the villi (cm)	Sample code
0.35	0.47	G1

Generally, water soluble nanoparticles might be able to be absorbed internally by wool [15]. The antibacterial properties of AgNPs mostly depends upon the size, pH and ionic strength of the medium [16]. The silver ions bind disulphide (S- S) and sulfhydryl (-SH) groups of wool fiber in the microbial cell wall's protein resulting in the disruption of metabolic processes, and ultimately cell death happen [17]. When silver molecules apply with binder or any cross-linking agent, an increased durability to clean wash was obtained in antimicrobial performance. Dyes obtained from plants have antimicrobial properties. Like beet root, pomegranate seeds, curcumin etc. due to the presence of tannins quantity. Other sources like naphthoquinones such as lawsone from henna, juglone from walnut and lapachol from alkanet exhibit antimicrobial activity [18].

In this study, the quantitative AATCC Test Method 1993-100 was used to evaluate the antibacterial activity against S. aureus and E. coli. The test method is described in our previous study [19].

Antibacterial test results in dyed sample and treated with 2 g/L nano silver and 0.5 g/L aluminum nano oxide and titanium nano dioxide (G2) and also, dyed sample and treated with 0.5 g/L Nano-silver, nano-aluminum oxide and nano-titanium dioxide (G9) were evaluated (Table 4).

In general, S. aureus is more resistant than E. coli because the cell wall membrane of S. aureus is thicker than E. coli. Result of sample in Table 4 shows that the small value of nanoparticles can be granted great antibacterial properties to samples especially against to gram-negative microbes. The antibacterial efficacy toward E. coli were higher than S. aureus at the low silver concentration, indicating that gram-negative bacteria are generally more sensitive to Ag NPs than gram-positive bacteria [20].

3.7. FTIR spectra analysis of samples

The FTIR spectra of raw wool, G1 and G2 samples are presented in Figure 9. The strong peaks at 2949 and 2867 cm^{-1} are attributed to CH_3 asymmetric/symmetric stretching vibrations, and those at 2916 and 2837 cm^{-1} are assigned to CH_2 asymmetric/symmetric stretching vibrations. The peaks around 842 and 810 cm^{-1} are due to CH_2 rocking vibrations in the crystalline regions [21].

Table 4: Investigation of antibacterial properties of treated samples.

Percentage reduction of S. aureus bacteria	Percentage reduction of E. coli bacteria	Number of S. aureus colonies	Number of E. coli colonies	Sample code
0	0	>1000	>1000	G2
30	100	700	0	G9

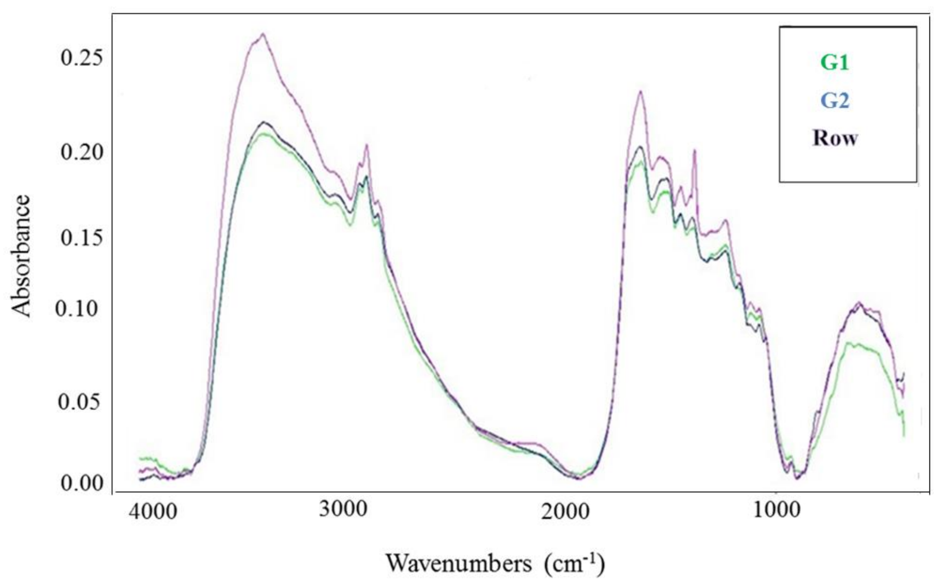

Figure 9: FTIR spectra of raw wool, G1 and G2 samples.

The main characteristic peaks of raw wool fibers due to protein macromolecule evidenced at 1639, 1546, and 1238 cm^{-1} correlated to amide I (C=O stretching), amide II (N–H stretching), and amide III (N–H in-plane bending and C–N stretching), respectively. Moreover, a broad band is considered at 3432 cm^{-1} due to the N-H stretching vibrations of terminal amino group. As can be seen, compared with raw wool sample, these bands' position and intensity undergo red-shift to lower frequencies and intensity in the dyed and treated wool fibers (G1 and G2), which is due to interaction of Ag nanoparticles with the amide groups [22].

4. Conclusions

In this study, firstly, woolen yarns were mordanted with alum, and then dyed with Walmut natural dye. After dyeing, carpet was woven and it was treated in three stages with three types of Nano-silver, Nano-titanium dioxide and Nano-alumina oxide. Using nano particles, the color strength of the samples has been reduced after receiving UV light, and self-cleaning properties of samples have been improved. The results showed that the hydrophilic properties and brightness values of samples is improved. SEM images indicated the effect of nano particles on treated carpets. The rate of water drop absorption of the treated samples has increased and their contact angle has decreased. In long-term static loading, the height is reduced by measuring the length of the villi. In the antibacterial test, the percentage of reduction against E. coli was higher than S. aureus bacteria. In the view of anti-odor test, an average of odor reduction was attained in the most samples. FTIR spectra of samples indicated the interaction of nanoparticles with amide groups. Present study displayed using of mentioned nanoparticles can reach multifunctional handmade carpet.

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