**INTRODUCTION**

Today’s ecological and economic responsibilities are directly affecting all stakeholders operating in the textile sector as well as in other sectors. At the same time, technological developments have led to changes in the expected characteristics of textile materials and development of different perspectives on material applications. Especially in recent years, instead of classical methods, it is preferred to use environmentally friendly and natural applications which allow lower cost, and energy use. This situation leads us to discussion of the classical processes aimed at achieving optimum use characteristics and benefit from textile products. Especially multifunctional textile products, approach the market share of conventional textile products gradually, and increasingly become preferred due to its high value added, different and attractive properties. For this reason, scientific researches on textiles are gradually shifting to different directions and investigating different application fields.

In recent studies on textile finishing processes, the use of the sol gel method has been particularly striking in alternative application methods. Properties and advantages of the chemical materials used by the sol gel method...
gel method can be transferred to the fabric. Using the sol gel method makes it possible to improve the fastness properties, the physical and chemical properties and the functional properties of the fabrics.

In the 1970s, the formation of monolithic inorganic jellies at low temperatures and conversion to glass materials brought the sol-gel method to the agenda [1]. The fact that the sol gel method can be carried out at low temperatures and the ability to handle the products in the desired shapes and formats make this method increasingly used by different scientific disciplines and engineering fields [2–3]. It is the front plan to investigate the usability of the sol gel method instead of different processes, especially in the scientific studies carried out in textile field.

Basically, sol gel method goes through this process: A metal alkoxide or metal salt are used within an acid/base-catalyzed suitable solvent as a precursor. Then, hydrolysis and condensation reactions occur consequently.

The factors affecting the sol gel reaction are pH, type and concentration of catalyst, type of solvent, type and structure of precursor, and temperature.

The aim of this study is to develop a new process that allows for the combined use of pigment printing and sol-gel technology using amorphous silica, colemanite and pumice, which are all provided by natural means [4]. It is aimed to develop the physical properties of cotton fabrics with the new process step created. Furthermore, thanks to the new process step, the fixing of the printing paddle has been carried out using sol gel technology instead of using of auxiliary chemicals (binder etc.); thus, ensuring the use of less chemical as possible.

MATERIALS AND METHODS

Materials

Scoured and bleached 100% plain weave cotton fabric (weight 284 g/m², 38 picks/cm, 21 ends/cm) has been used in this research. In preparing the sol gel solution; ethanol (C₂H₅OH, 96%, Merck, Germany) and distilled water (pH 7, conductivity 0.0182 mS/cm, 25°C) have been used as solvent. 0.1 N sodium hydroxide (NaOH, 100%, pellet, Sigma-Aldrich, Germany) has been used as the pH controller in the basic hydrolysis process. TEOS (Si(OC₃H₅)₄, % 98, Sigma-Aldrich, Germany) has been used as a precursor for sol gel solution.

In pumice, which is one of the rock types formed after acidic and basic volcanic activities, macro and micro scale pores are formed due to abrupt abandonment of the gases of structure during the formation and sudden cooling [4–8]. Due to the structure of micropores, which are usually interconnected, the pumice has very high thermal and sound insulation properties [4–5, 7, 9]. The hardness of the pumice rock is 5–6 according to the Mohs scale and chemically has up to 75% silica content.

Amorphous silica; volcanic and hydrothermal systems are sedimentary rocks with a generally white color, formed as silica sediments on the surface as a result of cooling off the surface and reaching saturation, as well as underground veins near neutral chlorinated waters, with fluids containing colloidal silica particles having a certain temperature and balance in solution [4, 10, 11]. Amorphous silica is in a structure that has free oxygen bonds due to the complete dissociation of Si-O bonds in spite of melting after 1730 °C. Through this feature; amorphous silica exhibits a low thermal expansion coefficient and is an important material for areas where thermal shock is required or where temperature changes require dimensional stability [12–13]. In addition, amorphous silica is an ideal refractive optical material due to its resistance to ultraviolet rays.

Boron is an element commonly found on earth. It is known that there are 230 kinds of boron element, which has boron oxide (B₂O₃) in different proportions, in nature [14]. Colemanite is the most commonly found boron minerals. Colemanite is a boron compound with crystalization properties in the monoclinc system. Colemanite is a primary boron derivative containing silica, calcium, boron, magnesium, iron and potassium oxides and having 50.8% B₂O₃ in its structure.

In this study, pumice was obtained from Isparta Gelincik region, amorphous silica from Keçiborlu-Isparta and colemanite from ETI Mining Corp. of Bigadiç Balkesir. All materials were sieved through number 200 sieve with a diameter of 0.076 mm. The physical and chemical compositions of materials are shown as in table 1.

Table 1

| Ingredients       | Unit | Pumice | Amorphous Silica | Colemanite |
|-------------------|------|--------|------------------|------------|
| B₂O₃ %            | -    | -      | -                | 40.00      |
| CaO %             | 4.68 | 0.31   | 27.00            |            |
| SiO₂ %            | 60.50| 92.48  | 4.00 – 6.50      |            |
| SO₄ %             | -    | -      | 0.60             |            |
| As ppm            | -    | -      | 35               |            |
| Fe₂O₃ %           | 3.38 | 0.09   | 0.88             |            |
| Al₂O₃ %           | 17.15| 2.60   | 0.40             |            |
| MgO %             | 2.09 | -      | 3.00             |            |
| SrO %             | -    | -      | 1.50             |            |
| Na₂O %            | 4.30 | 1.08   | 0.35             |            |
| SO₃ %             | 0.16 | 0.99   | -                |            |
| K₂O %             | 4.54 | 0.04   | -                |            |
| TiO₂ %            | 0.41 | 1.34   | -                |            |

Tubivis VP 681 (ammonium salt of anionic carboxylic acid derivate, CHT, Turkey) as a synthetic thickener and Tubiprint Fixer RE (nonionic melamine formaldehyde derivative, CHT, Turkey) as a fixerer have been
used to prepare pumice and colemanite added printing paste. Alginate SMT (anionic sodium alginate, CHT, Turkey) has been used as a thickener and Verolan TTY (ionic alkyl phosphonate mixture, Rudolf Duraner, Germany) has been used as an ion immobilizer for the preparation of the amorphous silica added printing paste.

EXPERIMENTAL MODELING METHOD

Printing recipes within the study are as follows.

| Solvent or Chemical       | Amount        |
|---------------------------|---------------|
| Pumice or Colemanite      | 25 / 50 / 100 g |
| Distilled Water           | X g           |
| Tubivis VP 681            | 17 g          |
| Tubiprint Fixerer RE      | 10 g / 1000 g |
| Amorphous Silica          | 25 / 50 / 100 g |
| Distilled Water           | X g           |
| Alginate SMT              | 40 g          |
| Verolan TTY               | 1 g / 1000 g  |

Printing of cotton fabrics have been carried out in accordance with the prescribed printing recipe and conditions [4, 15]. The printing process has been carried out in two rounds by using the prepared wooden printing templates. After printing stage, the fabric samples have been dried at 100 °C for 3 minutes. The conditioning of the finished printing samples has been applied under standard atmospheric conditions (65 ± 2% relative humidity and 20 ± 2 °C) for 24 hours.

After printing, wet weights were measured to ensure equal transfer of cotton and polyester fabric samples, and fabrics containing about the same value of printing paste have been taken as basis for the tests. The weight values of the cotton fabric samples after the approximate printing paste application are 440 g/m². The zeta potentials of all solid particulate materials (pumice, amorphous silica and colemanite) used in the printing process have been examined. When the zeta potential - pH graphs of pumice, colemanite and amorphous silica are examined, it is seen that all the materials in the basic region show common properties and have negative charges [4, 16, 17–18]. In this case, the sol gel solution has been prepared as basic catalyzed.

Sol gel recipe within the study is as follow:

| Chemicals     | Amount |
|---------------|--------|
| TEOS          | 11 ml  |
| Ethanol       | 50 ml  |
| Distilled Water | 35 ml |
| NaOH          | *      |

(* During the preparation of the sol gel solution, 0.1 N NaOH was added at a pH value of 9–10.)

Sol gel solution was stirred by magnetic stirrer at 25 °C for 30 mins and the process has been proceeded until the clear solution is obtained. The pH value of the obtained solution has been measured as 9.7.

After obtain of the sol gel solution, the cotton fabric samples have been coated with horizontal fulard at a rate 3.5 m/min under 2 bar pressure in 2 passages. Fixing of the coated fabrics has been carried out at 100 °C for 3 minutes and the fabric samples were conditioned under standard atmospheric conditions for 24 hours.

TESCAN MAIA3 XMU brand high-resolution scanning electron microscope has been used to display the surface morphology of the cotton fabric samples and to perform% elemental EDS point analysis. For the qualitative analysis of the samples, Shimadzu brand IRAffinity-1 model F-TIR device has been.

Fabric tensile strength and tear strength measurements have been carried out with the Lloyd LR5K Plus strength tester in accordance with TS EN ISO 13934-1 and TS EN ISO 13937–2. The flame retardancy tests which have been carried out using the 45° flame retardancy test, Bellmore, model TC-45 has been evaluated according to ASTM 1230-450. The measurement of the wear resistance of the fabric samples has been carried out in accordance with TS EN ISO 12945-2 using Nu-Martindale M 406 pilling and abrasion tester. The contact angle measurement of the samples has been made with the OCA 15 PLUS tester and the water permeability test has been carried out in accordance with the AATCC 42-2007 standard to measure water permeability values. Statistical evaluation of the test results applied to fabric samples has been evaluated via SPSS 18.0 Statistical Package Program.

RESULTS AND DISCUSSION

Surface morphology analyses

SEM photos of cotton fabric samples are given in figure 1. When SEM photos of fabrics are examined, it was observed that the structures are formed after printing process and sol gel process on fabric surfaces. The results of EDS analyses of cotton fabrics are given in figure 2.

When the EDS analyses results were examined, it was determined that the chemical components (Si, K, Al, Fe, Ca, Na, O, Ti, B, Ca) of all the additive materials (pumice, amorphous silica and colemanite) were present on the fabric surfaces. The F-TIR spectras of pumice and cotton fabric coated with pumice are given figure 3.

Figure 3 indicates the F-TIR ATR absorbance spectre of pumice and cotton fabric treated with pumice. The band between 3200 and 3800 cm⁻¹ are due to O – H stretching, between 3200 and 2800 cm⁻¹ are due to Al – OH stretching [4, 19, 20, 21, 22], While the peak is found at 1716 cm⁻¹ and the band between 1640
and 1690 cm\(^{-1}\) are due to C – O – C stretching [4, 20, 23]. The band between 1000 and 1110 cm\(^{-1}\) are due to Si = O and Si – O stretching [4, 15, 19, 21, 24]. Additionally, these peaks are between 900 and 1000 cm\(^{-1}\) are due Si – OH stretching and the peak at 742 cm\(^{-1}\) is due to Mg – Fe – OH [4, 20, 25].

The F-TIR spectras of amorphous silica and cotton fabric coated with amorphous silica are given figure 4.

Figure 4 indicates the F-TIR ATR absorbance spectre of amorphous silica and cotton fabric treated with amorphous silica. The band between 3200 and 3800 cm\(^{-1}\) are due to O – H stretching and between 2200 and 2400 cm\(^{-1}\) are due to C – H stretching [4, 19, 21]. The band between 1400 to 1690 cm\(^{-1}\) are due to COO\(^{-}\) and C = O stretching [4, 26, 27]. The band from 1000 to 1110 cm\(^{-1}\) is due to Si = O and Si – O stretching that caused a peak at 1038 cm\(^{-1}\). This is as a result of the presence of SiO\(_2\) in the amorphous silica structure [4, 15, 19, 21, 24]. Additionally, the peak at 786 cm\(^{-1}\) is due to amorphous silica and TEOS originated SiO\(_2\) compound [15, 21].

The F-TIR spectras of colemanite and cotton fabric coated with colemanite are given figure 5.

In figure 5, F-TIR analyses of colemanite and cotton fabric treated with colemanite are illustrated. As in all figures, the band between 3200 and 3800 cm\(^{-1}\) is caused by O – H stretching and between 2200 and 2400 cm\(^{-1}\) are due to C – H stretching [4, 19, 21]. The band between 1640 and 1690 cm\(^{-1}\) resulted from C = O stretching. The peak at 1654 cm\(^{-1}\) is due to H – O – H stretching and the peaks at 1558 and 1450 cm\(^{-1}\) are attributed to B\(_3\) – O stretching [4, 15, 28]. The peak at 1311 cm\(^{-1}\) and 1122 cm\(^{-1}\) are because of B – OH and B\(_4\) – O stretchings [29]. The band at 1000 to 1110 cm\(^{-1}\) are due to Si = O and Si – O stretching which caused peaks at 802 and 874 cm\(^{-1}\). This could be attributed to the existence of SiO\(_2\) in the amorphous silica structure and TEOS [4, 15, 21].

**Tensile strength and tearing strength values of cotton fabrics**

The tensile strength and elongation values of untreated and treated fabrics are given in table 2.
As seen in the values from table 2 the tensile strength of cotton fabric samples increased significantly after the printing and sol gel processes. When the table is examined in details, it is seen that the highest strength values are attained after 100 g/kg colemanite added printing and sol gel processing. When 100 g/kg colemanite was added into the printing paste and sol gel process was performed, the tensile strength increased by 41% in the warp direction and by 34% in the weft direction. After 100 g/kg of amorphous silica was added to the printing paste and sol gel process, the tensile strength increased by 32% in the warp direction and by 11% in the weft direction. After 100 g/kg pumice added process; the warp direction tensile strength value was increased by 35% and the weft direction tensile strength was increased by 16%. It is also seen that the values of tensile strength are increased in parallel with the addition ratio for all materials.

When an untreated cotton fabric is subjected to a force parallel to the fiber axis, the macromolecules in the fiber structure and the crystallites are formed resulting to a reaction against cohesive forces and the force tends to break when the force reaches a value above this effect. Rupture occurs in the oxygen bridge connecting the macromolecules. After the printing paste and pumice, amorphous silica and colemanite structures on the fabric samples, the resistance of the fabric samples to the breaking forces increased resulting to the increment of the tensile strength value [4, 15].

As seen in the values from table 2 the tensile strength of cotton fabric samples increased significantly after the printing and sol gel processes. When the table is examined in details, it is seen that the highest strength values are attained after 100 g/kg colemanite added printing and sol gel processing. When 100 g/kg colemanite was added into the printing paste and sol gel process was performed, the tensile strength increased by 41% in the warp direction and by 34% in the weft direction. After 100 g/kg of amorphous silica was added to the printing paste and sol gel process, the tensile strength increased by 32% in the warp direction and by 11% in the weft direction. After 100 g/kg pumice added process; the warp direction tensile strength value was increased by 35% and the weft direction tensile strength was increased by 16%. It is also seen that the values of tensile strength are increased in parallel with the addition ratio for all materials.

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The elongation values of the cotton fabric samples after the pumice, amorphous silica and colemanite added printing and sol gel coating processes have lower values than the untreated fabric samples. The reason for this result is that a new and rigid structure is formed on the surface of the fabric with natural based material, thickener and sol gel layer after printing and sol gel coating process [4, 15].

The values of the tensile strength and elongation obtained after the processes with all materials were evaluated with One Way Anova test. As a result of the analysis made, the sigma value for both warp and weft direction was found to be 0,000. After the homogeneity of the variance, the sigma value is calculated as 0,000. This result indicates a heterogeneous distribution of variances. It is determined that there is a significant statistical difference between all the processes after the analysis using the technique Tamhane’s T2 post hoc.

The tearing strength values of untreated and treated fabrics are given in table 3.

When the table is examined, the highest values in tearing strength are attained after 100 g/kg of colemanite added processes as well as in tensile strength values. After 100 g/kg colemanite addition process, an increase of 27% in the warp direction and 23% in the weft direction were obtained.

The results show that the highest tensile and tearing strength values for all samples are obtained after colemanite added processes. The reason for this result is the B based new bonds (B – OH, B 4 – O) formed in the colemanite structure. These bonds are the result of B 2O 3 compound, which is 40% in the structure of colemanite.

UV resistance values of cotton fabrics

Textile materials have different reflectivity and absorption rates of sun UV radiation. Fibers with low UV absorption properties, such as cotton fabrics are considered to be more risky than fibers containing polymers with high absorption properties [30, 31].

In this study, transmittance values of the fabrics were measured in UV A (315–400 nm) and UV B (290–315 nm) regions with 5 nm wavelength intervals according to AS/NZS 4399-1996 standard. The results obtained after measurement are given in figure 6. The results obtained from UVA and UVB measurements of cotton fabrics are given in table 4. When the obtained data in table 1 are analyzed, it is seen that the cotton fabric samples have the lowest % transmittance values in the UVA and UV B regions after the amorphous silica added printing and sol gel process. The highest UV protection values for cotton

![Image](https://via.placeholder.com/150)

Fig. 6. % Transmittance values of cotton fabrics
fabric samples after the process added amorphous silica are due to the refractive optical material property of the amorphous silica. The reason is that the hydroxides (OH\(^-\)) present in amorphous silica can absorb some wave lengths well and absorb Si – O vibration at high intervals [4, 32].

Air permeability values of cotton fabrics

The air permeability values of the cotton fabric samples are given in table 5.

|                  | Amount (g/kg) | Air permeability (L/m²/s) |
|------------------|--------------|--------------------------|
| Cotton fabric    |              |                          |
| Cotton fabric treated with pumice | 100 | 52.66                    |
|                   | 50           | 55.01                    |
|                   | 25           | 58.41                    |
| Cotton fabric treated with amorphous silica | 100 | 11.71                    |
|                   | 50           | 12.86                    |
|                   | 25           | 13.06                    |
| Cotton fabric treated with colemanite  | 100 | 65.87                    |
|                   | 50           | 68.84                    |
|                   | 25           | 71.33                    |
| Cotton fabric    |              | 202.38                   |

Table 5

According to table 5 air permeability values of all cotton fabrics is decreased as a results of processes. The lowest air permeability values were attained after amorphous silica added printing and sol gel process. This can be explained by the density of SiO\(_2\) bonds formed and the natural alginate thickener used. In contrast to the pumice and colemanite addition processes using synthetic thickeners, a denser layer is obtained on the surface of the amorphous silica-added fabric after the use of a natural thickener with a high solids content.

One-way ANOVA analysis of the obtained air permeability values revealed that the resulting sigma value was 0.000 and that all the procedures had a statistically significant difference. This proves that all processes with additive materials are effective on the cotton fabric.

Flame retardancy values of cotton fabrics

The flame retardancy values of cotton fabric samples after pumice, amorphous silica and colemanite-added printing and sol gel processing are given in table 6.

|                  | Amount (g/kg) | Ignition time (s) | Yarn breakage time (s) |
|------------------|--------------|------------------|------------------------|
| Cotton fabric    |              |                  |                        |
| Cotton fabric treated with pumice | 100 | 22.62            | 57.82                  |
|                   | 50           | 21.82            | 56.34                  |
|                   | 25           | 18.22            | 53.62                  |
| Cotton fabric treated with amorphous silica | 100 | not burnt       | not burnt              |
|                   | 50           | not burnt        | not burnt              |
|                   | 25           | not burnt        | not burnt              |
| Cotton fabric treated with colemanite  | 100 | 26.36            | 76.4                   |
|                   | 50           | 25.72            | 75.32                  |
|                   | 25           | 23.7             | 73.6                   |
| Cotton fabric    |              | 15.64            | 34.92                  |

Table 6

When the test results are examined, it is seen that the samples of cotton fabrics have nonflammable properties after amorphous silica-added processes. This is explained by the non-flammability properties of the sodium alginate based thickener used in the amorphous silica printing recipe. In the same test, after the colemanite addition process, the flammability properties of the fabric samples increased and the burning times increased by 220% compared to the untreated fabric sample. Due to the B\(_2\)O\(_3\) and MgO compounds found in the colemanite structure, it exhibits a high non-flammable characteristic [4, 15].

Wear resistance values of cotton fabrics

% Weight changes of fabric samples after 5,000, 10,000 and 20,000 cycles were measured for the measurement of wear resistance of cotton fabric samples. The obtained results are given in table 7. When the table is examined, it is seen that cotton fabric samples treated with amorphous silica have the lowest% weight loss value after 20,000 cycles. The highest wear resistance was attained after amorphous silica added printing and sol gel processing because of the formation of a denser and more valid structure on the surface of the natural based thickener welded fabric. In addition, when the table is examined, it shows that the wear resistance of all fabric samples that are processed in compared to the non-treated fabric sample is increased.

The contact angles and water permeability values of cotton fabrics

The contact angle measurement results of cotton fabric samples after pumice, amorphous silica and colemanite addition printing and sol gel coating process are given in the table 8. Water permeability measurement results of cotton fabric samples after pumice, amorphous silica and colemanite addition printing and left gel coating process are given in table 9. When the obtained contact angle and water permeability results are examined, it is seen that the contact angle and water permeability values are improved.
CONCLUSIONS

In the study, the changes in the physical properties of cotton fabric printed with pumice, amorphous silica and colemanite and fixed with sol gel method were investigated. As a result of the obtained data, it is concluded that all the processing steps are effective on the tensile and tear strength, air permeability, flame retardancy, UV protection, wear resistance properties of cotton fabrics. Morphological and elemental analyses have been proven to be successful in transferring the chemical compounds of natural basis materials used in the new process to cotton fabric samples. In this study, several finishing processes (tensile strength, tear strength, air permeability, flame retardant, UV protection) were applied simultaneously using completely natural based materials. In addition, an example in textile has been created for the applicability of the sol gel process, which is developing and widespread in many fields. Because it uses resources effectively, it is more eco-friendly and the fact that it has lower energy cost due to lower fixation temperature; sol gel method in textile has been closely examined. It has also been proved in this study that it will be a prominent source for future studies of researches in this field.

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