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Deposition of CdO Semiconductors on yarns by Dip Coating Method and Gas Sensor Applications

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ABSTRACT

Thin films produced by deposition of metal oxides on different surfaces show semi-conductor properties that are sensitive to the surrounding atmosphere components. The sol-gel dip coating technique, which is simple, accessible, adjustable, and repeatable based on desired parameters, is widely used in the production of CdO thin films. In this study, CdO metal oxide thin films were coated on polyamide, acrylic and cotton yarns by sol-gel dipping method in three different molarities of 0.1 M, 0.3 M and 0.5 M starting solutions. Structural properties of CdO thin film coated yarn samples were investigated using SEM and EDX analyzes were performed. The sensor tests of the yarn samples for LPG gas were carried out in the specially designed gas sensor measurement system and in the gas chamber. The 0.5 M CdO thin film coated cotton yarn samples showed better semiconductor properties and gas response than the other samples.

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KEYWORDS

Yarn Gas Sensor, Semiconductor, CdO, Metal-Oxide, Thin Film

INTRODUCTION

Charge carriers on the surface of a semiconductor are sensitive to the composition of the surrounding atmosphere [1]. The material surfaces are coated with different semiconductor oxides such as SnO_2 [2], CuO [3] and ZnO [4] in order to apply semiconductor properties. CdO metal oxide has low electrical resistivity [5], wide band gap [6], high gas sensing [7] and optoelectronic properties [8]. Therefore, CdO is used in photodiodes [3], photovoltaic cells [9], electrodes [10] and gas sensors [11].

CdO metal oxide thin films deposited by SILAR method [12], spray pyrolysis method [13], sol-gel spin coating method [14], [15], magnetron spray method [16], thermal evaporation method [17] and sol-gel dip coating methods [18] on different surfaces. Because of its ease of synthesis [19], effective process control [20], and ability to produce highly homogeneous film solutions [21], the sol-gel method is widely used [22]. Dip coating technique is preferred in CdO thin films with the advantages of low installation cost [23], requiring less energy [24], allowing different surfaces to be coated, being simple [25], economical [26] and fast. At the same time, re-dipping process is an effective method for creating a homogeneous surface for fibrous structures [27]. CdO films are used in N_2 gas sensors [28], H_2O_2 sensors [29], LPG, butane/propane [30], [31] sensors applications.

Studies in the last ten years were searched in the Web of Science database with the title "CdO Thin Films" and it was seen that 249 publications. It has been observed that these publications received 3190 citations in total. The bibliometric network map of the distribution of publications

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by keywords is shown in Figure 1. The analysis results showed 555 keywords in the studies within the scope of the subject in the VOSviewer program. Words repeated at least 3 times, synonyms and irrelevant words were removed. Thus, a network map was created by 46 different keywords.

Examining the network map, CdO thin films were produced by chemical vapor deposition, sputtering and commonly Sol-gel coating methods. Generally, the surface morphology, structural, electrical and optical properties of these films were investigated. CdO films have been used in different structures such as gas sensors, optoelectronic materials and solar cells.

In this study, dip coating technique was preferred as a method to form of the CdO thin film. Nm 6 acrylic, Ne 2 cotton and 105 tex polyamide yarns were chosen as the surface to be coated. The surface properties of three different yarns prepared at different concentrations were analyzed using Field Emission Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (FESEM-EDX). Gas tests were carried out by assembling CdO coated yarns to a programmable control card specially designed to detect the gas released into the medium.

MATERIAL AND METHOD

Cadmium acetate dihydrate ($Cd(CH_3COO)_2 \cdot 2H_2O$) (Sigma-Aldrich, ≥98.0% purity) salt was used as a reagent, 2-methoxyethanol ($CH_3OCH_2CH_2OH$) (Sigma-Aldrich, ≥99% purity) as solvent and monoethanolamine ($NH_2CH_2CH_2OH$) (Sigma-Aldrich) as stabilizer in the experimental process. Cadmium acetate dihydrate salt was added to 100 ml of 2-methoxyethanol for 3 different concentrations (0.1 M, 0.3 M and 0.5 M). The sol-gel solutions were stirred continuously for 2 hours with a magnetic stirrer at room temperature by adding 2 drops of monoethanolamine. The solutions were rested for 24 hours and made ready for deposition.

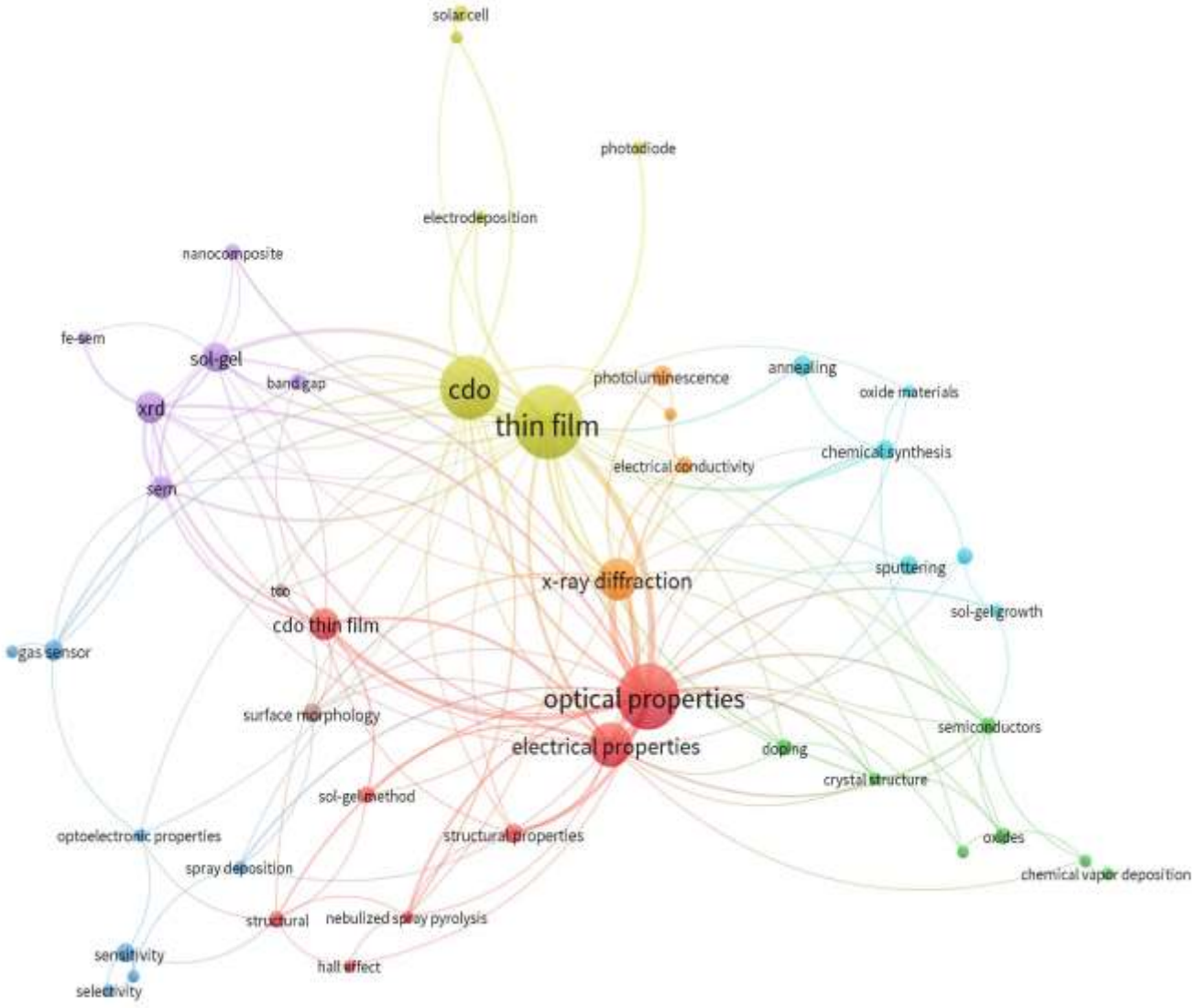

Figure 1. Bibliometric network map by keywords of publications in Web of Science database

Metal oxide thin films were deposited on the surfaces of selected natural and synthetic yarns. Widely used in the textile industry, cotton from natural fiber group yarns [32], [33], polyamide [34] and acrylic yarns [35] from synthetic fiber group yarns were selected. It is foreseen that the deposition on different yarn volumes and yarn surfaces will show different adhesion properties. It was thought that the cotton yarn with staple structure would absorb more solution than the acrylic and the polyamide yarns with filament structure.

Sample yarns cut in equal sizes were cleaned with deionized water and dried at 85°C. They were deposited by dip coating method in solutions of different concentrations. During the coating process, the yarn samples were dipped in the solutions for 1 minute each time. It was dried at 150 °C for 2 minutes. The dip coating process was tested in 5, 10 and 15 repetitions. In the 5-layer coating process, the surfaces of the samples were not completely covered in a homogeneous manner. It was determined that there were excessive accumulation and debris on the sample surfaces in 15 layers of coating. In the 10-layer coating, it was observed that the yarn surfaces were coated homogeneously and without clumping. 10 layers of coating was determined as the optimum number of coatings. The yarn samples, whose dipping and drying processes were finished, were annealed at different temperatures for their crystallization. When the temperature was exceeded 300 °C for annealing, deterioration and burning occurred in the structures of all yarn samples [36]. It was observed that the CdO structure did not crystallize on the surfaces of the yarn samples when the annealing process was analyzed below 150 °C. At first, the samples were annealed at 3 different temperatures as 150 °C, 200 °C and 250 °C for 1 hour. According to the literature review, it has been seen that the annealing temperature of CdO thin films is in general between 200-450°C for crystallization to occur on the surface. In addition, the crystal structure and semiconductor properties improved with the annealing temperature increase from 200 °C to 450 °C in literature [37], [38], [39]. However, the optimum annealing temperature was determined as 200 °C in order not to harm the cotton yarn and to form the CdO crystal structure. For these reasons, the annealing temperature was applied as 200 °C and 1 hour throughout the study.

RESULTS AND DISCUSSION

The morphological properties of the CdO-deposited yarn samples were analyzed by the Field Emission Scanning Electron Microscope (ZEISS Supra 40VP FESEM). CdO structure is generally observed in irregular morphology and cauliflower shape [40], [41], [42] in the literature.

The differences in particle sizes were caused by the dipping cycle, the difference of sample yarn surfaces and solution concentrations. The surface images of 3 different sample yarns at 3 different solution concentrations were examined. Yarn Sample Series are shown in Table 1.

Polyamide, acrylic and cotton yarn samples were coated with CdO metal oxide by dipping in the sol-gel solution prepared at 0.1 M concentration, respectively. SEM images after annealing are shown in Figure 2. According to A1 and A2 images, the coating did not adhere to the yarn surface and crystal structure was not formed. In B1, B2, C1 and C2 images, it was observed that adhesion started on the sample surfaces, but the amount of coating was insufficient.

SEM images of polyamide, acrylic and cotton yarn samples with 0.3 M concentration of CdO deposited are shown in Figure 3. In A3 and A4 images, the coating did not form properly on the surface and gave an appearance of plastering. Coating was started to occur according to B3 and B4 images. However, crystallization, the predicted cauliflower shape and a homogeneous structure did not occur. Although, cauliflower-shaped crystallization occurred on the surface of the samples in the C3 and C4 images, it was determined that the solution concentration was not sufficient and the surface was not completely covered in a homogeneous film.

Table 1. Yarn Sample Series

Series	Sample	Series	Sample	Series	Sample
A1-A2	0.1 M Polyamide	A3-A4	0.3 M Polyamide	A5-A6	0.5 M Polyamide
B1-B2	0.1 M Acrylic	B3-B4	0.3 M Acrylic	B5-B6	0.5 M Acrylic
C1-C2	0.1 M Cotton	C3-C4	0.3 M Cotton	C5-C6	0.5 M Cotton

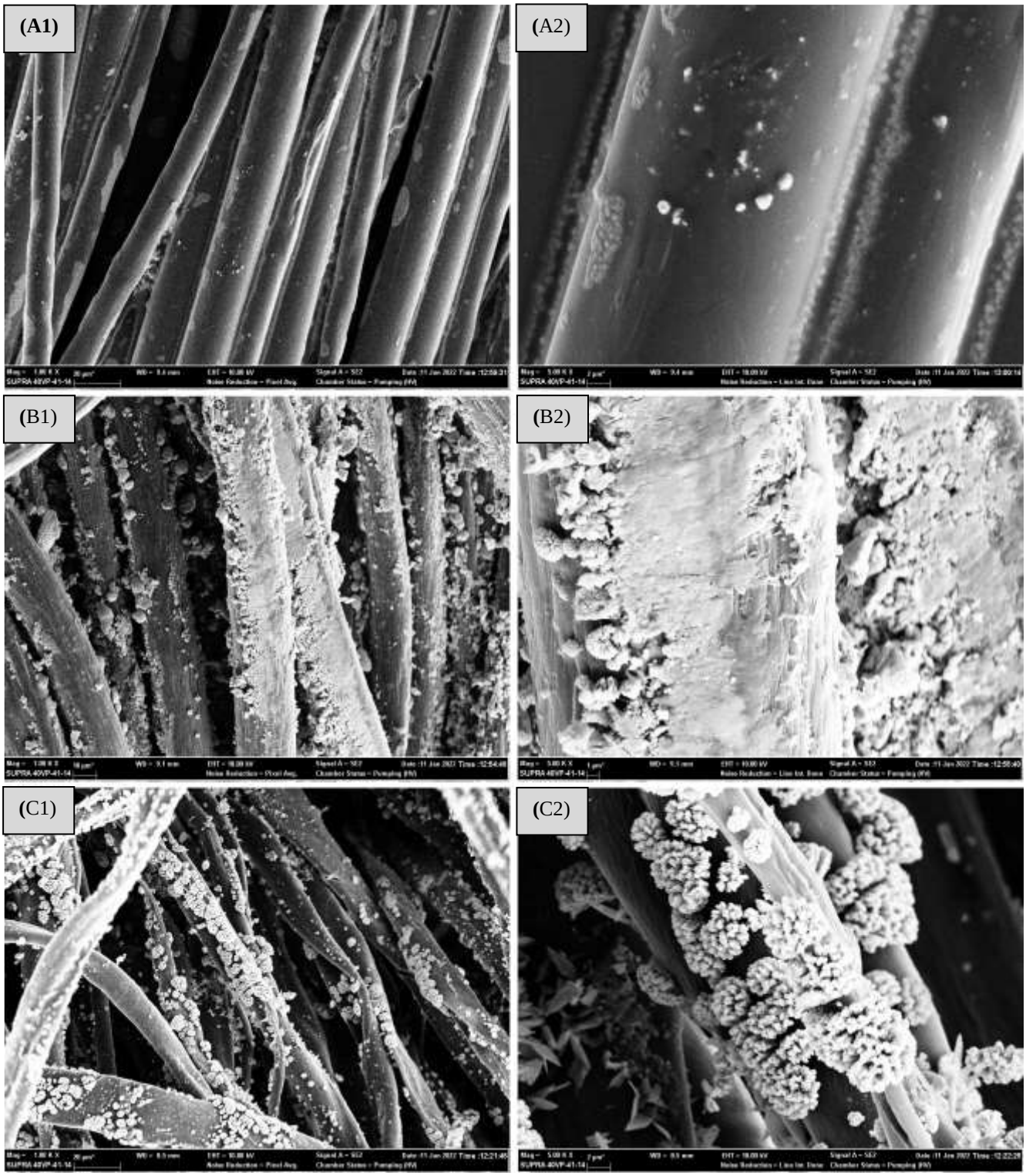

Figure 2. SEM images of the deposition of 0.1 M CdO coating solution on (A) polyamide yarn, (B) acrylic yarn, (C) cotton yarn surfaces

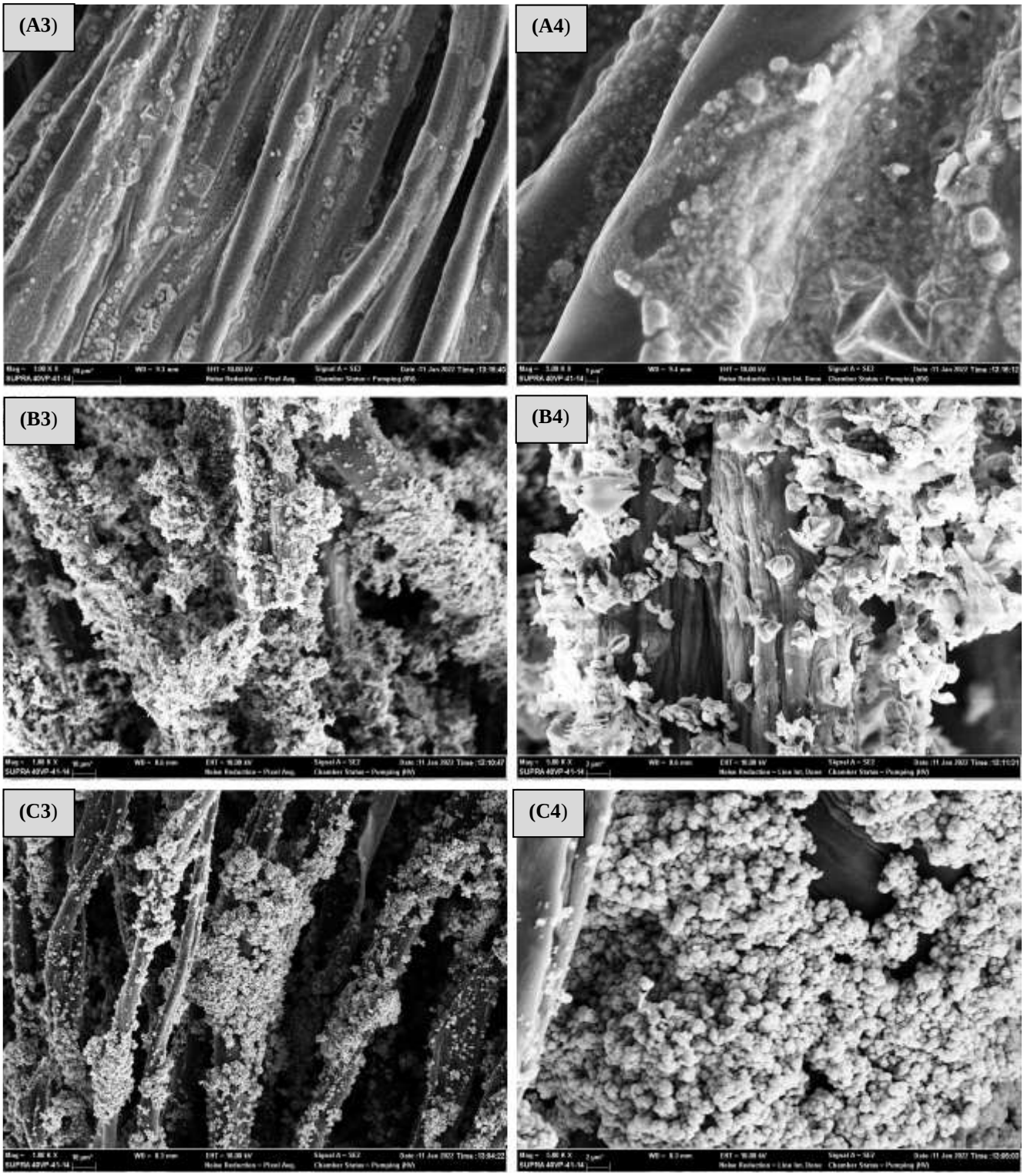

Figure 3. SEM images of the deposition of 0.3 M CdO coating solution on (A) polyamide yarn, (B) acrylic yarn, (C) cotton yarn surfaces

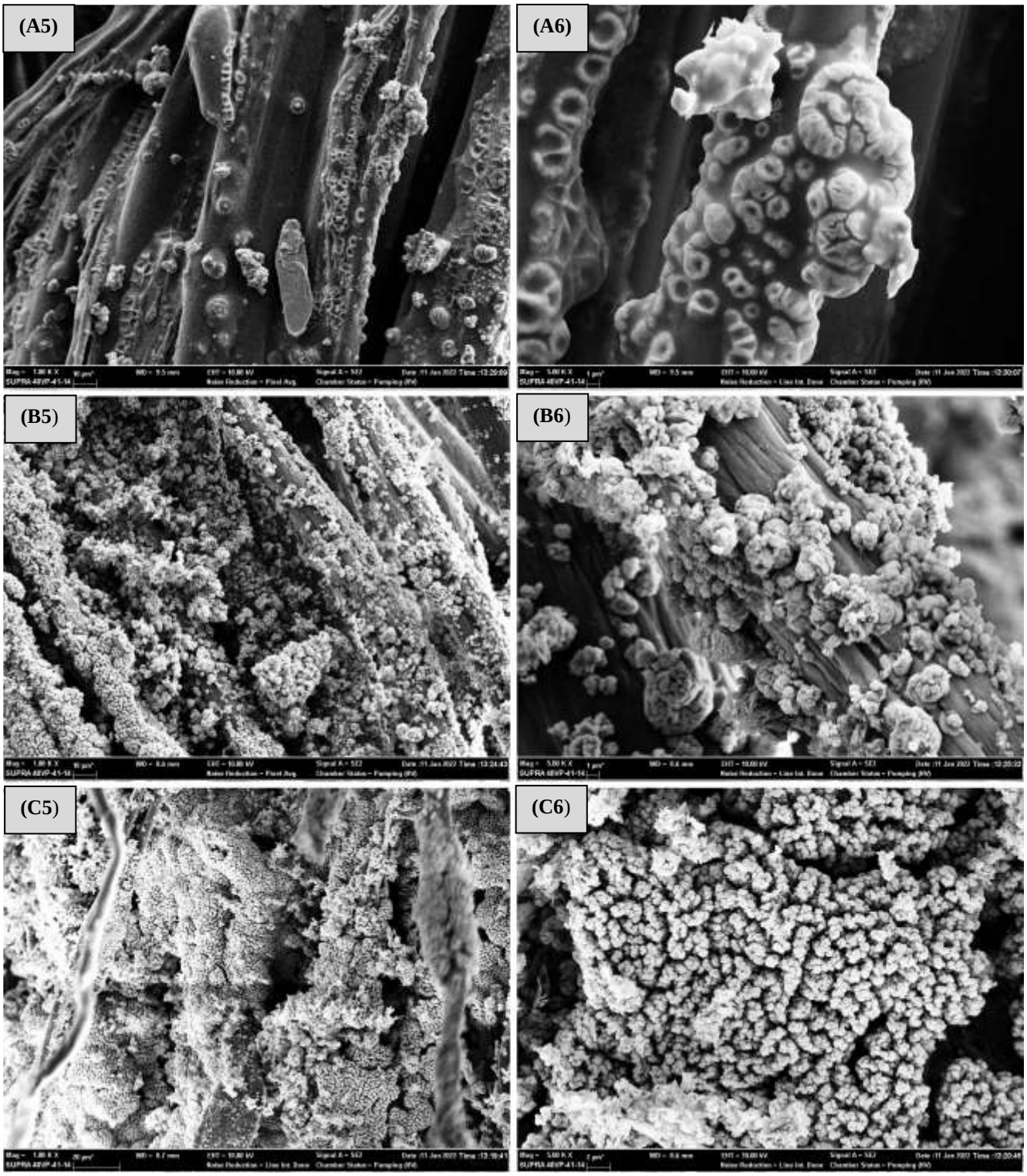

Figure 4. SEM images of the deposition of 0.5 M CdO coating solution on (A) polyamide yarn, (B) acrylic yarn, (C) cotton yarn surfaces

SEM images of polyamide, acrylic and cotton yarn samples with 0.5 M concentration of CdO deposited are shown in Figure 4. According to A5 and A6 images, CdO particles were formed but did not crystallize on the sample surfaces. Deterioration in the yarn structure and surface abrasions began to occur with the increase in solution concentration. The sample surface vitrificate because of the annealing temperature increased. The increase in the solution concentration caused the deterioration of the yarn surface. B5 and B6 images showed that the metal oxide structure was partially crystallized. The cauliflower-shaped crystal structure specific to CdO was observed. The deposited film did not show semiconductor properties due to the obvious gaps on the surface. According to the C5 and C6 images, the film is almost homogeneously distributed over the entire surface. It was observed that the CdO particles both

crystallised on the surface in the form of cauliflower and that the particles adhered closely and well. Therefore, it has been confirmed that the cotton yarn sample covered with CdO thin film can be used as a flexible and semiconductor material.

The weight changes of 0.1 M, 0.3 M and 0.5 M 10-layer CdO thin film coated cotton yarns are shown in Table 2. While the weight of the section of 0.1 M cotton yarn was 0.052 g, it was 0.063 g after 10 times re-dipping. After annealing for 1 hour at 200 °C, its weight was measured as 0.053 g. In other words, there was a weight gain of approximately 21% after the coating process. After the annealing process, there was an increase in weight of about 2% compared to the initial state. The initial weight of 0.3 M yarn was measured as 0.049 g. After the coating process, it became 0.083 g with an increase of approximately 70%. After annealing process, it was measured as 0.077 g and weighted approximately 57% more than its original state. The initial weight of 0.5 M yarn sample was measured as 0.050 gr. This sample showed the highest percentage increase in weight after coating and annealing processes. It was measured as 0.141 g with an increase of 182% after coating process. After annealing process, it became 102% heavier than its initial state and was determined as 0.101 g.

The amount of adhesion of the coating on the surface increased with the increase of the concentration. In addition, the amount of absorption of CdO particles in the solution of the yarn sample increased. The density of CdO particles in solution increased because of the concentrations increased. CdO particles were more attached to the surface of the cotton yarn and spread homogeneously because of the staple and fibrillar structure of cotton yarn.

Metal oxide surfaces adsorb oxygen in the normal medium, changing electron mobility on the surface. When there are different gases in the medium, metal oxide semiconductor materials interact with the gases, reducing the amount of oxygen adsorbing. This situation causes a resistance change [43], allowing metal oxide semiconductor materials to be used for gas detection purposes.

Table 2. Change in weight of cotton yarn sample by thin film coating

Weight / Sample	Before Deposition	After Deposition		After Annealing	
	g	g	%	g	%
C1	0.052	0.063	21.15	0.053	1.92
C2	0.049	0.083	69.40	0.077	57.14
C3	0.050	0.141	182	0.101	102

The electronic circuit diagram of the yarn gas sensor produced is shown in Figure 5. Gas sensor electronic circuit; +5V DC voltage, R1 and R2 resistance used by changing depending on the structure and internal resistance of the yarn gas sensor, Analog Port (A1), which detects the voltage change between 0-5 V depending on the gas type, amount and contact time in the medium, Digital port 1 (D1) and Digital port 2 (D2), which transfer information to the stimulating systems (Led, Buzzer, etc.) according to the determined parameters, Ground (GND) to complete the circuit, it consists of a programmable Micro Controller Card (MCC), which provides analog-digital information conversion and control, to which the entire system is connected.

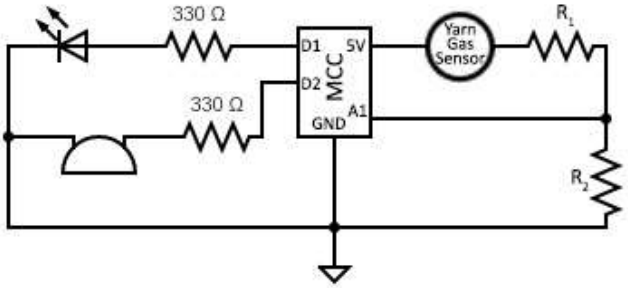

Figure 5. Gas sensor electronic circuit diagram

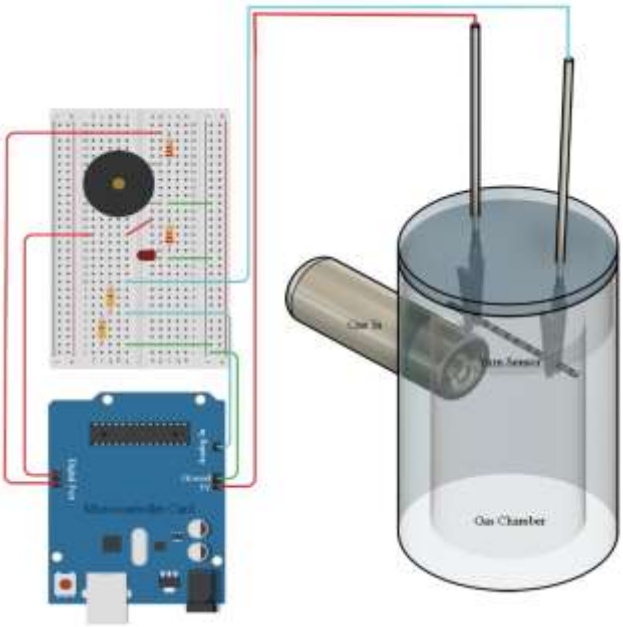

Figure 6. Yarn gas sensor (YGS) measurement system

The gas sensor measurement system designed specifically for the semiconductor yarns produced within the scope of this study is given schematically in Figure 6. Gas detection tests were performed on this system. The chamber shown and modeled in the figure was produced from a 3D printer using a transparent filament. +5V voltage was given from MCC to YGS in the system. The current completed the circuit by passing through the yarn gas sensor and then the resistors R1 and R2. LPG gas was given to the chamber in which the yarn sensor was located and the sensor reacted with the gas. The voltage difference values on the resistors R1 and R2 are received from the A1 port. The amount of

gas supplied to the system changed depending on the time. For this reason, the intensity and duration of the reaction also changed. These data received from the A1 port were converted into digital values and transmitted to the Leds and Buzzers. As the amount of gas in the gas chamber increased, the contact of the thread gas sensor with the gas and the voltage variation on the resistor also increased. This situation increased the brightness of the light in the LED and the sound level of the Buzzer. Likewise, light and sound levels also decreased as the amount of gas decreased. The stimulators can be active when the amount of gas in the medium reaches a certain level, and passive when it falls below, by defining certain parameters in the system.

5V constant current was given to the yarn sensor samples in the gas sensor measurement system we designed. The semiconductor properties of the yarn samples were determined. The polyamide yarn sample did not respond at all to the given voltage. Acrylic yarn samples responded irregularly. However, it was thought that the measurements were not sufficient for the sensor applications. Cotton yarn samples responded regularly. For this reason, it was decided to conduct gas sensor trials with cotton yarn samples. Gas sensor measurements were applied as 10 repetitions. The data of the sensor measurements are shown in Figure 7. First of all, a constant voltage was applied to cotton yarn samples showing semiconductor properties, and the voltage on the resistor was balanced for 30 seconds in normal room condition. During this time, 4.22-4.39 V data was received on the resistor from the system. It was waited for 30 seconds in the fixed interval where the voltage on the semiconductor yarn was balanced. LPG gas was given to the gas chamber from the 60th second. Gas molecules in the medium reduced the amount of adsorbed oxygen on the surface of cotton yarn samples with CdO deposited. This caused the voltage change necessary for the gas to be detected and the sensor to respond. The voltage change on the resistor was measured between 2.56-3.09 V for 60 seconds when there was gas in the medium. When the gas inlet given to the medium was removed at 120th seconds, the measured values returned to the values close to their initial state. This situation continued in a balanced way for 60 seconds. After 180th seconds, gas was given to the medium for the second time. Similar to the first situation, values between 2.70 and 2.94 V were taken from the system. This cycle was applied with 10 repetitions in 60-second periods. In the sensor measurements made when the gas was removed from the medium in the last period, the voltage change returned to values close to 3.84 V. The average of 10 repetitive measurements was calculated. While the voltage value was about 4.09 V in the absence of gas in the medium, it was about 2.78 V in the presence of gas. It was observed that the measurement values decreased gradually at almost every stage. The final values is slightly lower than the initial values. This can be thought of as the increase in the amount of gas adsorbed on the surface of the yarn as the gas presence time in the medium increases. Although the voltage values decreased, they did not decrease significantly and also the acquired semiconductor property did not changed. For this reason, the repeatability of the yarn gas sensor has a good level.

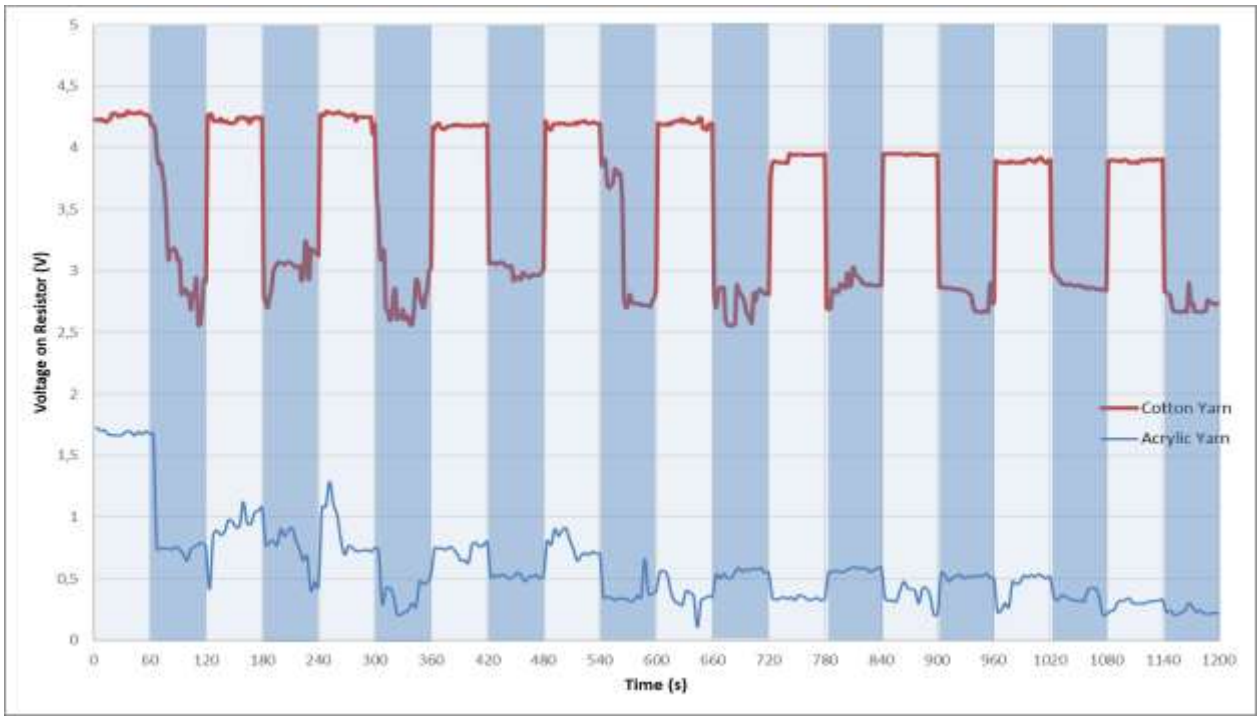

Figure 7. Yarn gas sensor applications

CONCLUSION

CdO was chosen as the metal oxide because it is easy to apply. Sol-gel dip coating technique was preferred because it is reproducible according to the desired number of layers and it is an economical method. Acrylic, cotton and polyamide yarn surfaces, which are actively used in various textile products are coated with thin films to produce semiconductor yarns. Experiments were made at different solution concentrations, number of coating layers and annealing temperature. Optimum semiconductor property was determined in cotton yarn sample at 0.5 M CdO concentration, 10 times re-dipping and 200 °C for 1 hour annealing temperature. It was determined that the polyamide yarn sample did not withstand the annealing temperature, glassification occurred on the surface, and therefore it was not suitable for deposition of the CdO thin film by dip coating. It was observed that the acrylic yarn sample showed semiconductor properties under the determined optimum conditions, but it was not sufficient for gas sensor applications. As an alternative to expensive, complex systems, the gas chamber and gas measurement system are originally designed. The designed, simple to use, low-cost gas measurement system has enabled gas sensors produced with different methods and materials to be tested with various gases. Looking at the gas measurement data, it is clearly seen that the produced semiconductor cotton yarn can be used as a light, flexible and portable gas sensor. It is possible to produce a gas sensor from cotton yarns and use it as a material with high added value.

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