Synthesis, characterization, and coating application of a highly conductive polyaniline-TiO₂ nanocomposite

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Abstract

In the present work, synthesis, and characterization of a highly conductive polyaniline (PANI) doped with titanium dioxide (TiO₂) nanocomposite that allows static charges to flow through the floor and be safely grounded has been reported. The effect of different dopant concentrations on the conductivity and other properties of the PANI nanocomposite was investigated. Fourier Transform Infrared shows the characteristic vibrational bands of N–H, C–H, C=C, aromatic ring, and C≡N at 3428, 2913, 1566, 1466, and 1294 cm⁻¹ of the PANI and v(Ti–O) at 470 cm⁻¹ of its composites. The lattice strain examined by Rietveld analysis of radiography diffraction data exhibits microstrain broadening of 7.3 due to local deformation of Ti and O atoms caused by interaction with PANI. Transmission electron microscopy analysis of the average size and distribution of PANI and its composite particles confirms the dispersion of TiO₂ in the PANI matrix, with diameters ranging from 2 to 22 nm and a median of 7 nm. The conductivity of PANI was increased with increasing the dopant of TiO₂ up to 10% (wt/wt) which was the optimal one exhibiting an excellent value of 33.93 S/cm, for the first time. PANI doped with 10% TiO₂ (PAT10) is a promising material for electronic manufacturing facilities, clean rooms, and other environments where static electricity can be a hazard. Highly conductive PAT10 nanocomposite as a floor coating effectively reduces electrostatic risks, providing both safety and durability.

Keywords: Conductive coating, Electrostatic discharge, Electrostatic discharge coating, Polyaniline/TiO₂ nanocomposites, Polyaniline

1. Introduction

Electrostatic discharge (ESD) is a natural physical phenomenon that can cause fires and explosions in industrial sites, leading to significant damages and losses. ESD occurs when there is a sudden flow of electrons from one surface or human to another, creating a miniature spark that can damage electronics and cause data loss. ESD is caused by the accumulation of static electricity between two surfaces due to friction, contact, and separation. Prevention and control of ESD are essential to avoid damages and losses. A protective ground is necessary to provide a traceable path to the ground and prevent static from generating on people. Anti-static floors and conductive materials can be used to prevent ESD and increase the electrical conductivity of epoxy compounds. However, the creation of a layer of epoxy resin that contains conductive materials on the surface of a floor is widely used to prevent static electricity buildup and electromagnetic interference problems. Conductive materials transfer charges from people’s feet to the ground, effectively grounding the charges and removing them from the area. To increase the electrical conductivity of the epoxy matrix, electrically conductive materials such as copper, silver nanowires, carbon nanotubes or fibers (CNTs/CNFs), and conductive polymers (CPs) such as poly (3, 4-ethylenedioxythiophene) (PEDOT) and poly (pyrrole) (PPY) are added to the resin. Materials used for ESD shielding can be classified into conductive and dispersive materials based on their resistance ranges. Conductive materials have a volumetric resistivity of less than 104 Ω/
cm² or a surface resistivity of less than 105 Ω/cm², while static dispersive materials have a volume resistivity of 104–1011 Ω/cm or a surface resistance of 105–1012 Ω/cm². Polyacrylonitrile (PAN) is a conductive polymer that is commonly used in coating systems, particularly in corrosion protection. The conjugation system on PANI's backbone makes it a preferred conductive polymer in many applications. However, the conjugation alone is not sufficient to make the polymer conductive, and additional electrons or holes must be injected into the material. PANI is also often combined with inorganic nanoparticles such as TiO₂ to create nanocomposites with unique properties, including different electrical, optical, and mechanical properties. PANI is a p-type semiconductor with a band gap of 2.1 eV, and TiO₂ is a n-type semiconductor with a band gap of 3.13 eV. Therefore, the two types of particles can be linked together by a p-n junction, and the resulting PANI/TiO₂ nanocomposite can provide barriers to electronic migration.

This study investigates the effect of dopant concentrations on the conductivity and properties of a PANI nanocomposite. The highly conductive PAT10 (PANI/TiO₂) nanocomposite was used in epoxy resin formulations and the antistatic properties which have potential applications as a barrier to electronic migration were investigated.

2. Experimental

2.1. Material

Aniline, Ammonium persulfate (APS), and TiO₂ with an average particle size of 150 nm were used as a pigment in this study. Calcium carbonate with an average particle size of 200 nm was used as an extender. The dispersant Alkylammonium salt of a high molecular-weight copolymer with a molecular weight of 5000 g/mol was supplied from Witting and Disspers (W and D) BYK 9076 (Germany). An industrial grade Xylene was employed as a pollutant. Epoxy resin EPOTEC YD 128 (Germany), Epoxy hardener ANCAMINE 1618 as Curing Agent (Germany), reactive diluent EPOTEC RD 108 (Germany), Surface Additive BYK 306 (Germany), and anti-foaming agent BYK A 530 (Germany), Leveling agent BYK 320 (Germany), HCl (33%), Methanol, and Acetone.

2.2. Synthesis of polyaniline

The template-free method was used to prepare the PANI nanostructure as in the previous article but with the molar ratio of the oxidant increased to 1.25 of the monomer ratio to reduce the presence of unpolymerized aniline and to obtain the highest yield of PANI and reached 92%, compared 60% of the previous study. Aniline hydrochloride monomer was prepared by mixing 1 mol of aniline with 1 mol of HCl in an aqueous solution. The solution was stirred for 1 h to ensure the formation of a homogeneous solution of aniline–HCl. The weighted APS was dissolved in deionized water and slowly added to the reaction mixture in a dropwise manner under vigorous stirring using a magnetic stirrer at room temperature (22 °C) for 2 h. Then, the mixture was allowed to polymerize by stirring for 24 h. The color of the solution gradually changed from white to green. Then, a dark green precipitate is obtained by filtering the mixture. The precipitate was washed repeatedly with distilled water, dilute hydrochloric acid, methanol, and acetone to remove any unreacted regents and left to dry at room temperature. PANI hydrochloride powder was finally obtained by slight grinding.

2.3. Synthesis of polyaniline/TiO₂ composites

The synthesis of PANI-TiO₂ composites involved an in situ chemical polymerization method. A solution of aniline hydrochloride (6.5 g PANI + 6 ml HCl (33% v/v)+100 ml deionized water) served as the base for the synthesis. TiO₂ was introduced at varying concentrations (2%, 4%, 6%, 10%, 12%, and 14% by weight of aniline hydrochloride). To achieve a uniform dispersion of TiO₂ in the monomer solution, vigorous stirring was carried out for 30 min. Subsequently, a solution containing 14.3 g of ammonium persulfate in 100 ml of 0.1 N HCl was added to initiate the polymerization process. The yield percentage of the polymerization process ranged from 88% to 90%, with an increase in the ratio of incorporated TiO₂.

2.4. Preparation of coating formulations

A 40 g PANI composite TiO₂ 10% (PAT10) (selected for coating formulation due to the highest conductivity) was added to a portion of 100 g epoxy resin containing the 1 g BYK 9076 wetting and dispersing agent. The ingredients are mixed well with a mechanical stirrer until a homogeneous
slurry is obtained. Then the conventional additives such as an anti-foaming agent 2 g BYKA 530, leveling agent 0.8 g BYK 306 and BYK 320, and 57 g CaCO₃ were added to the mixture while stirring continuously for 10 min these component A was mixed with Component B of 50 g ANCAMINE 1618 by mixed ratio 100:25% and was applied to the concrete substrate for evaluation. Note that: the PAT10 was added in proportions of 0, 10, 20, 30, and 40% of the total composition and was applied to the concrete substrate for optimization and evaluation.

3. Result and discussion

3.1. FTIR analysis

FTIR spectroscopy was used to characterize the chemical structure and functional groups of PANI and PAT10 materials. The FTIR spectrum of PANI as shown in Fig. 1, typically exhibits strong stretching vibrational bands of N–H, C–H, C–C, aromatic ring, and C–N at 3428, 2913, 1566, 1466, and 1294 cm⁻¹, respectively. Moreover, the bands observed at 1132, and 792 cm⁻¹ are corresponding to aromatic C–H in the plane and out-of-plane bending vibrational modes, respectively.

In addition, the PANI-doped TiO₂ (PAT10) shows characteristic absorption bands corresponding to the functional groups present in both the PANI and the dopant. The most characteristic absorption bands in the FTIR spectra of TiO₂ are typically seen in the range of 400–1000 cm⁻¹ and it has been reported around 450 cm⁻¹. As a result of first derivatives of IR spectrum, the vibrational band at 470 cm⁻¹ in the of PAT10 is related to the Ti–O stretching vibration. In addition, the absorption bands in the range between 400 and 1000 cm⁻¹ were shifted to a lower wavenumber by about 2 cm⁻¹. This finding confirms the presence of the dopant of TiO₂ as physically interacted with PANI.

3.2. Structure analysis

XRD technique was used to determine the structural properties and crystal structure of PANI and PAT10 composite. Fig. 2a illustrates the XRD pattern of PANI in comparison with the PAT10. The results show the amorphous or semi crystalline structure of PANI and formed relatively weak peaks at 2θ around 20.03 and 25.36° caused by the polymer chains being arranged parallel and closely spaced from one another. The XRD pattern of PANI doped with 10% TiO₂ (PAT10) shows diffraction peaks corresponding to the crystal structures of both the PANI and the dopant. The XRD pattern of PAT10 shows the characteristic peaks of the anatase phase of TiO₂ (JCPDS PDF#021-1276) at 2θ equal to 27.44°, 36.09°, 39.19°, 41.23°, 54.05°, 56.64°, 62.74°, 64.04°, 65.48°, and 69.01° which corresponding to (110), (101), (111), (211), (220), (002), (310), (221), and (301), respectively. The relative intensities and positions of the diffraction peaks of PAT10 were further analyzed using Rietveld refinement as shown in Fig. 2b. The tetragonal crystal system of TiO₂ (Space group: P42/mmm #136, a: 4.5933 Å, c: 2.9592 Å) was used as initial crystallographic parameters for Rietveld analysis. The diffraction peaks of PAT10 were well-indexed and matched with the literature and no extra peaks of impurities were found. However, the lattice parameter ‘a’ was a little increased to 4.5946 Å by about 0.0013 Å. In contrast, the lattice parameter ‘c’ was a little decreased to 2.9588(2) Å by about 0.0006 Å, which is not significant chemical interaction could occur. Overall, these results suggest a physical interaction (Van-der-Waals Forces) between TiO₂ and PANI. Furthermore, a size-strain plot based on the Williamson-Hall method was used to assess crystal size and lattice strains as shown in Fig. 2c.

The PAT10 exhibits microstrain broadening of 7.3, and it would be attributed to a slight local deformation of Ti and O atoms caused by interaction with PANI. Meanwhile, the crystallite size of PAT10 was equal to 38.46 nm, which confirms the nano-composite formation of PANI TiO₂ doped materials.

3.3. Morphology analysis

Scan electron microscopy (SEM), transmission electron microscopy (TEM) and statistical analysis
were used to investigate the shape and morphology of the PAT10 composite at the nanoscale. It worth to mention, high-conductive materials tend to accumulate electrons from the SEM beam, leading to charging effects. This can cause distortion and artifacts in the images, making it difficult to obtain accurate information about the sample’s surface for both PANI and PAT10 as shown in Fig. 3a–d. However, the TEM image of the PAT10, and PANI composites as shown in Fig. 3b–e shows that the PANI is distributed throughout the TiO2 matrix in the form of nano, spherical-like structures in which PANI is shown as a core covered by a TiO2 layer. The degree of dispersion of TiO2 in the PANI matrix can be statistically determined by measuring the average size and distribution of the PAT10 particles (diameters in the range of 2–22 nm with a median of 7 nm) compare with (diameters in the range of 5–30 nm with a median of 18 nm) of PANI, which confirm PAT10 nanocomposite as shown in Fig. 3c–f.

3.4. Optoelectronic analysis

The electronic properties of PANI and PAT10 were investigated using diffuse reflectance spectroscopy, at room temperature. It is well known that diffuse reflectance of a material is a measure of how much light is reflected by the surface of the material when it is illuminated by a light source. Thus, the
diffuse reflectance of PANI can vary depending on the specific properties of the material, such as the degree of crystallinity and the presence of impurities or doped TiO₂. In general, PANI is known to have a relatively low diffuse reflectance, which is typical of many semiconducting materials. This means that a relatively small fraction of the light that is incident on the surface of the material is reflected, and most of the light is absorbed. Fig. 4 depicts the Kubelka-Munk reflectance spectra of PANI in comparison with PAT10 materials. The x-axis intercept of a linear extrapolation of the low energy region of each curve was used to calculate the indirect bandgap (Eg) values.

It worth to mention, the pristine TiO₂ nanoparticles band gap was reported 3.2 eV in literatures. However, the band gap of PANI TiO₂ composite could be calculated based on the higher absorption band of the curve which equal 2.9 eV, and that is lower than pristine TiO₂. In addition the results show a clear significant red shift up to 1.21 eV compared with nondoped PANI materials which could be a result of interstitial state or excitons due to formation of PANI TiO₂ composite. This finding suggests that PAT10 exhibited a bit stronger response in the visible light range than PANI, which could boost conductivity efficiency in different applications.

Fig. 4. (a) The optical band gap energy of polyaniline and PAT10; (b) TGA and DrTGA of PAT10; (c) Plot of ln[-ln(1-x)] versus 1000/T of four steps decomposition (b).
However, it’s been reported that PANI doped TiO2 samples can have a higher diffuse reflectance than pure PANI samples due to the increased crystalline structure of PANI in the presence of TiO2 as discussed above in the XRD section.

3.5. TGA and thermodynamic analysis

Thermal gravimetric analysis (TGA) conducted in this study was used to measure the weight loss of the material as a function of temperature. PAT10 nanocomposite was analyzed through TGA/DTgTA (differential thermal gravimetric analysis) with a heating rate of 10 °C min⁻¹ in the range of 30–1000 °C, under N2 gas, to show the thermal degradation of PANI-doped TiO2 and better understand the mechanism of this process as shown in Fig. 4b and c. Thus, PAT10 undergoes thermal degradation at temperatures 50–145 °C, resulting in a weight loss of around 3% due to dehydration and removal of adsorbed water molecules on the surface of PAT10 nanoparticles. The result accords with the evaporation of moisture trapped inside the polymer or bound to the polymer backbone, as demonstrated by the degradation stage in the second step (229–350°C). In addition, the thermal degradation of PAT10 is breaking down the polymer material when exposed to high temperatures greater than 500 °C weight loss of 27% from the initial weight. TGA can be used to determine the kinetics and thermodynamic parameters of PANI-doped TiO2. Moreover, the Coats-Redfern model is a method used to analyze the TGA data, which allows the determination of various thermodynamic parameters such as activation energy, entropy, enthalpy, and Gibbs free energy, which are recorded in Table S1 (in the supplementary file). The activation energy is an important parameter that represents the energy required to initiate the degradation process. The results of this study show that the activation energy increases as the temperature increases, indicating that it becomes harder to initiate the degradation process at higher temperatures. The activation energy of 28011 J/mol at 145 °C and 34943 J/mol at 650 °C, are relatively high values, which suggest that the thermal degradation process is difficult to initiate and it requires a lot of energy to start. This can be an indication that the PAT10 has higher thermal stability at 650 °C than at 145 °C. Even that the PANI doped with 10% TiO2 (PAT10) give a relatively good stability lower than 100 °C which more than enough for our target application. The entropy of the system is a measure of the disorder or randomness of the system. The results of this study show that the entropy of the system decreases as the temperature increases, indicating that the system becomes more ordered as the temperature increases. The entropy values of −245 J/mol K and −266 J/mol K at 145 and 650 °C, respectively, suggest that the thermal degradation process leads to a decrease in disorder in the system. Indeed this behavior could be associated with an increase in crystallinity and ordered arrangement of TiO2 particles within the polymeric matrix. The thermal degradation process might influence the arrangement of these components, leading to changes in their crystalline structures. The enthalpy is a measure of the heat absorbed or released during the degradation process. The results of this study show that the enthalpy increases as the temperature increases, indicating that more heat is absorbed as the temperature increases. The enthalpy values of 24827 J/mol and 28499 J/mol at 145 and 650 °C, respectively, suggest that the thermal degradation process absorbs a lot of heat. This can be an indication that the polymer has a high heat of combustion. Finally, Gibbs's free energy is a measure of the energy available to do work. The results of this study show that the Gibbs free energy increases as the temperature increases, indicating that the energy available to do work also increases as the temperature increases. The Gibbs free energy values of 119007 J/mol at 145 °C and 235091 J/mol at 650 °C, suggest that the thermal degradation process releases a lot of energy, which can be used to do work. Overall, the results suggest that the PANI-doped TiO2 is thermally stable, releases a lot of energy when degraded, and has a high heat of combustion.

3.6. Conductivity measurements of PANI and PANI/TiO2

Conductivity is a crucial property of materials. It was determined for PANI and its composites with TiO2 through the four-probe technique as shown in Figure (a). The results revealed that the pure PANI exhibited a conductivity of 9.12 S/cm, a value that was subject to improvement through the incorporation of TiO2. The conductivities of PANI doped with TiO2 were studied with different weight ratios, namely 2%, 4%, 6%, 10%, 12%, and 14%. The results showed a remarkable increase in conductivity with increasing TiO2 content up to 10%, with values of 25.33, 26.56, 27.43, 33.93, 19.09, and 13.36 S/cm, respectively. It was intriguing to observe the influence of TiO2 on the conductivity of PANI. The values of conductivity observed with different ratios of TiO2 showed a fluctuation, with the significant conductivity of 33.93 S/cm observed with 10% TiO2 content (Table S2 in the supplementary file). However, the
conductivity showed a decline with a further increase in TiO2 content, suggesting a delicate balance between the two components in optimizing the conductivity. Furthermore, it is interesting to note that the increase in conductivity with the increase of TiO2 content up to 10%. This could be attributed to several factors related to the incorporation of TiO2 nanoparticles. TiO2 nanoparticles can act as electron acceptors and donors, facilitating charge transfer between the polymer chains. This increased charge carrier mobility can enhance the overall conductivity of the composite. Also, the combination of PANI and TiO2 may exhibit synergistic effects, where the properties of the composite are greater than the sum of its individual components. This synergy can arise from the complementary electronic properties of the two materials.

3.7. Antistatic performance

Epoxy coating is a well-known insulation coating, with very high surface resistivity. When PAT10 is incorporated with 40, 30, 20, and 10 wt% of Epoxy, the EP-PAT10 presented good electrical properties. Table 1 shows the measured surface resistivity and conductivity which indicate that the EP-PAT10 can dissipate a charge by both surface conduction (according to ANSI/EIA-541 standard). Compared with pure Epoxy without PAT10.

The basis of the antistatic coating process is the free transfer of electrostatic charges along with the polymer matrix within the coating system, causing the film to become electrostatically dispersive. Numerous elements, including the type of epoxy resin, the form, the quantity, and the dispersion of CFs, as well as the characteristics of additives, affect a coating’s antistatic properties. The term ‘percolation threshold’ refers to the minimum volumetric concentration of CFs that must be present to create an electrostatic conductive network. Thus, and according to the obtained antistatic results, we can say that the percent of 20% will be considered the beginning of the percolation threshold point for the present formulation as reported in.

3.8. Measurement of surface humidity

The results obtained with the Frequency-based Moisture Meter (FFM) 100 device on a concrete cube, an epoxy-coated concrete cube, and an epoxy-PAT10 coated concrete cube showed results of 52, 48, and 28% moisture. These results show a very sharp decrease in moisture content in a coating containing PAT10 compared with the epoxy-only coating and the uncoated concrete cube. This sharp decrease is attributed to the greater hydrophobicity of the PANI/TiO2-containing Epoxy coating. The drastic hydrophobicity can be explained as follows: Despite the relative hydrophilicity of epoxy resin, PANI as well as titanium oxide individually, the mixing of these three compounds results in hydrogen bonds between the active sites represented by the oxygen atom in the epoxy resin, the hydrogen atom in the doped acid in PANI and the oxygen atom in TiO2. This association results in the absence of active sites in the Epoxy-PANI/TiO2 capable of making hydrogen bonds with water molecules in moisture.

3.9. Measurement of adhesion

As mentioned above, the load is increasingly applied until the dolly is pulled off. The force required to pull the dolly off or the force the dolly withstands is recorded as the adhesion force. Failure occurs along the weakest plane within the system comprised of the dolly, adhesive, coating system, and substrate, and will be exposed by the fracture surface. The result of the adhesion strength test was completely different from the usual one, as the paint consisting of epoxy with PANI oxide - titanium showed a very high adhesion strength, which resulted in breaking the upper layers of the concrete cube without separating the paint layer, which means infinite adhesion between the paint and the concrete surface. Error! Reference source not found. (b) shows the degree of adhesion of the paint material and the refraction of the cube without separation of the paint layer.

3.10. Abrasion resistance

Table S3 (in the supplementary file), and Fig. 5c show the relationship between the number of cycles and the lost weight of the tapered rotary scraper, which was performed by ASTM D 4060. Although the pristine epoxy showed a weight loss of 88 mg, we note the Epoxy-PAT10 20%, lost only 28 mg after 500 cycles, which means a superior improvement in abrasion resistance. It is generally considered an

| Table 1. PAT 10 Add ratio with Surface Resistivity and Conductivity of Epoxy. |
|-----------------|-----------------|-----------------|
| Add ratio       | Surface Resistivity $\Omega$/cm² | Conductivity S.cm² |
| EP- Blank       | $1.9 \times 10^{13}$ | $526,315 \times 10^{-16}$ |
| 10% PAT 10      | $18.44 \times 10^{11}$ | $542.29 \times 10^{-15}$ |
| 20% PAT 10      | $5.42 \times 10^{9}$ | $184.50 \times 10^{-12}$ |
| 30% PAT 10      | $0.3 \times 10^{8}$ | $333.3 \times 10^{-10}$ |
| 40% PAT 10      | $0.1 \times 10^{6}$ | $1 \times 10^{-5}$ |
excellent performance result. To our knowledge, no one has been able to achieve this result previously. This means that the Epoxy coating with PAT 10 was able to withstand a great deal of wear without showing signs of wear or deterioration. The positive direction in the abrasion resistance may be attributed to the incorporation of the nano PANI-TiO2 particles into the epoxy coating which increased the interface surface interaction between the nano-sized PANI-TiO2 particles and epoxy coating base matrix providing more compact, more smooth, and less abraded film as compared with the coating without PANI-TiO2 particles.45 Investigations using potentiodynamic polarization measurements, cure durability, ultraviolet immovability, and abrasion resistance of polyamine-cured ilmenite epoxy coating for oil and gas storage steel tanks in the petroleum sector were studied by Al-Sabagh and colleagues that show the improvement in the abrasion resistance character may be described as previously,46 the well-dispersion of composite particles tend to occupy holidays such as pinholes and voids in the thin-film coating and serve as the bridges in the interconnected matrix, causing a reduction in the total free volume and an enhancement in cross-linking density of the cured film. As such, the cured epoxy ilmenite coating has reduced chain segmental motions and improved stiffness.

3.11. Conclusions

In conclusion, the study reports the synthesis and characterization of highly conductive PANI doped with TiO2 (PAT10) nanocomposite and using it in Epoxy coating formulation for ESD applications. The conductivity of PANI was found to increase with increasing the dopant up to 10% (wt/wt), resulting in an excellent value of 33.93 S/cm. TEM analysis showed that the particles of PAT10 have diameters ranging from 2 to 22 nm and a median of 7 nm. The incorporation of nano PANI-TiO2 particles into the epoxy coating (Epoxy-PAT10 20%) results in a remarkable enhancement of abrasion resistance, demonstrated by a weight loss of only 28 mg after 500 cycles, compared with 88 mg for the pristine epoxy. We suggest that PAT10 is a promising material for environments where static electricity can be a hazard and that it could be used as ESD coatings as like as Intensive care rooms in hospitals, floors of chemical and flammable materials stores, roofs and floors of ships and oil tankers … etc. The percentage of adding PAT10 to the epoxy paint is a 20% percolation threshold, which can then overcome the resistivity of the epoxy coating and turn it into a dissipative coating. It is also possible to add larger amounts of up to 40% if more conductivity is desirable.

Authors contributions

Khaled A. Altannyhi: Conceptualization, Data curation, Investigation, coding, Formal analysis, Methodology, Writing—original draft, Formal analysis, Supervision. Elsayed M. Elnaggar/ Mohamed M. Elsenety Data curation, Formal analysis, Methodology, Investigation, Writing—original draft, Investigation, Methodology. Badr A. Elsayed: Writing—review & editing, Supervision.

Conflicts of interest

None declared.

Acknowledgements

Authors acknowledge support from the Department of Chemistry, Faculty of Science, Al-Azhar University.
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