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Research article

Preparation and characterization of conductive blends of polyaniline with polyphenol red

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Abstract

Polymers are widely employed in biomedical applications, pharmaceutical product formulation, and drug delivery systems. Since every polymer has its own distinct properties, polymer blends will have novel chemical and physical properties. Functionally, the purpose of blending polymers is to improve, customize, or maximize material performance. In this study, polyaniline and polyphenol red polymer mixtures were prepared electrochemically and characterized with XPS imaging and SEM whether their distribution was homogeneous. The mixture of aniline and phenol red was deposited glassy carbon electrode (GCE) surface using the cyclic voltammetry technique in the potential range of -0.80 V to 2.00 V with 50 mV/s scan rate for 25 cycles. The phase separation of the two polymers was demonstrated by a combination of spectroscopic imaging and microscopy. For this purpose, the X-ray spot size and step number were set to 50µm. 1 x 1 mm^2 area scan of the polymer mixtures was performed, and spectra were obtained at each pixel in an array of 20 x 20 pixels. Chemical imaging was obtained by applying Principal Component Analysis (PCA) to collected XPS survey spectra. For the morphological characterization, scanning electron microscopy (SEM) was employed, and images were obtained at magnifications of 5000 x. The results obtained in the mixtures prepared with 5%, 10% and 25% were better compared to the mixture prepared with 50% polyphenol red. Since the X-ray spot size is limited, the desired image resolution could not be obtained. It was shown that XPS imaging studies could also be used for examining the distribution of different and unknown polymer mixtures together with SEM.

Keywords: *Chemical imaging; polyphenol red; polyaniline; polymer blend; principal component analysis; X-ray photoelectron spectroscopy*

1. Introduction

A true revolution in the field of polymers, conducting polymers are significant and novel materials. They have potential use in a variety of fields. Polyaniline (PANI) looks unique thanks to its exceptional electrical conductivity, biocompatibility, and lack of toxicity. PANI has some surprising properties, but its low solubility and processability have prevented it from being used in any practical applications so far. To overcome these limitations PANI-based composites, polymer grafting, and polymer blends have been developed (Pina and Falletta, 2022; Ghovvati et al., 2023; Morais et al., 2023; Nasir et al., 2023; Shokrollahi et al., 2023). All conductive blends of polymers (CPs) are prepared using two primary methods: chemical synthesis and electrochemical synthesis. The first method typically produces powders, but the second allows for the production of films. While chemical synthesis seems to be the best route for the large-scale production of CPs, the resulting polymers are difficult to work with and require structural modifications to improve their electronic properties. Conversely, conducting polymers represent a revolution in the field of polymers, since they represent an important and

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innovative class of materials. They qualify for employment in numerous industries. Polyaniline (PANI) is distinguished by its high electrical conductivity, biocompatibility, and low toxicity. Despite its outstanding characteristics, PANI has not yet found practical uses due to its poor solubility and processability. Electrochemical control of the polymerization process is more efficient, notwithstanding its ineffectiveness for large scale manufacturing (Li et al., 2012; Ogundele et al., 2023). PANI is composed of repeating aniline units. The presence of alternating -NH- groups and aromatic cycles provide compounds with distinct oxidation states. In contrast, phenol red (PR) is the usual pH indicator in many cell and tissue culture media since it allows for a rapid assessment of the culture's health. PR has also been utilized in several techniques to detect cellular hydrogen peroxide and human peroxidase enzyme activity (Morgan et al., 2019). The structures aniline and phenol red are presented in Fig.1.

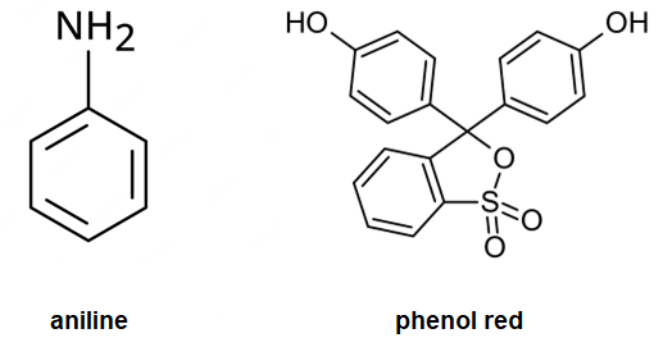

Fig. 1. Chemical structures and images of aniline and phenol red.

PANI can be used in a variety of biomedical contexts, including as an antioxidant, as an agent with antimicrobial and antivirus activity, as a drug delivery system, as a cancer therapy, and as biosensors etc. (Gizdavic-Nikolaidis et al., 2004; Boomi et al., 2014; Isakova et al., 2016; Zhao et al., 2017; Li et al., 2018; Liang et al., 2018; Ahmadkhani et al., 2019; Minisy et al., 2021; Ghovvati et al., 2023; Riaz et al., 2023). There is an ever-increasing demand for the creation of novel and more effective materials that can deliver drugs to the organs and tissues that are being targeted. One of the most important considerations relates to the requirement that there should not be an excessive amount of drugs or an insufficient amount of drugs in order to achieve a steady and well-controlled release of drugs over a specific amount of time (Pina and Falletta, 2022). Smart drug delivery systems are a promising tool in this regard because they integrate pharmaceutics, materials science, molecular biology, engineering, and other disciplines. The support can either actively contribute to the drug release mechanism or sit on the sidelines and play the role of an inert carrier. Among the various supports, CPs hold a significant position due to the extraordinary capabilities they possess, which are primarily associated with their one-of-a-kind characteristics, such as electroconductivity, pH sensitivity, light-sensitivity, and so on.

In very recent years nanofiber composites consisting of chitosan and PANI have been fabricated by in situ chemical polymerization (Minisy et al., 2021). Ketoprofen was able to be encapsulated within the composites due to the free volume space that the composites provided. It was demonstrated that the drug release is dependent on the pH of the medium, and that the release increased as the pH increased. On the other hand, working with a simulation of biological fluid yielded some encouraging results. By taking advantage of the sensitivity of PANI-based materials to changes in pH, a pH-electroactive bacterial cellulose/PANI hydrogel that allows for the controlled release of drugs was developed (Li et al., 2018). Chemotherapy for late-stage cancer is emerging. Accurate release control enhances drug accumulation in a specific target tissue in alternative cancer treatment. You et al. (2017) developed trastuzumab-modified PANI-mediated polymeric nanoparticles for tumour cell uptake. This was carried out to look into the possibility of a cis-platinum delivery that could be precisely controlled. CPs are utilized as transducers in biosensors due to the extraordinary properties that they possess, which include electroconductivity, redox properties, biocompatibility, and high levels of sensitivity. These properties allow for the CPs to detect changes in their environments with a high degree of accuracy. Shoaie et al. (2019) comprehensively discussed on PANI and PANI composite biosensor applications. Recent advances in PANI-based biosensors necessitate their use. Electrical stimulations are responsible for the regulation of most the human body's control mechanisms. In addition, electroactive materials have been extensively researched for potential applications in tissue engineering, with the goals of stimulating cellular responses and enhancing tissue regeneration. The properties of pure PANI are not suitable for use in any application involving tissue engineering.

The employment of a single polymer does not always meet the site-specific and time-controlled release profile requirements of a sophisticated drug delivery system, according to scientific literature. Even though polymers of various origins and of varying chemical natures have been introduced in biomedicine as carriers for the purposes of drug delivery, this remains the case. Polymer blends, from simple physical mixtures to more complex core-shell strategies and even polymeric block copolymers, will enable site-specific and rate-controlling drug delivery in pharmaceutical sciences. In spite of the fact that there is no one simple recommendation that can be applied in every situation, it would be helpful to apply various strategies that are relevant to the blending of polymers in order to overcome formulation limitations (Ghasemiyeh and Mohammadi-Samani, 2021; Barker et al., 2023; Yazie et al., 2023). Methods for blending polymers can lead to the formation of possible molecular interactions between the blended polymers, each of which has its own set of characteristics. The synthesis procedure needs to be improved to overcome cytotoxicity, processability, and physicochemical properties that prevent the clinical use of PANI and PANI-derivatives. When homopolymers are combined through the process of polymer blending, the resulting product may have altered physicochemical properties in comparison to those of the homopolymers by themselves. The production of miscible polymer mixtures is the primary benefit that comes from blending different types of polymers. Blended polymers' physical properties depend on both their homopolymer properties and their intramolecular interactions. Blending polymers can create a new product with a second functionality that can interact with other polymers and drugs (Jones et al., 2005).

In a wide variety of contexts, the surface properties of polymer blends are of critical importance. Because of the surface segregation of the component with the lowest surface energy, the physicochemical properties of polymer blends can be drastically different at the surface than they are in the bulk. In this work, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) have been used to

characterize surface and bulk properties since the surface properties and morphology of the prepared polymer blends directly affect their functionality.

2. Materials and methods

2.1. Chemicals and reagents

All reagents were of analytical reagent grade. Phenol red (PhR) ($C_{19}H_{14}O_5S$), aniline ($C_6H_5NH_2$) which is distilled two times for purification, perchloric acid ($HClO_4$), alumina (Al_2O_3) was supplied from Sigma Aldrich, Merck and BDH chemicals. All solutions were prepared using ultrapure water which produced from Millipore Milli Q system (18.2 Ω). All electrochemical experiments were performed at room temperature.

2.2. Preparation of polymer blends

GCE was polished alumina powders, rinsed and ultrasonicated in water prior to voltammetric measurements. Electrochemical cleaning was performed by keeping GCE in 0.1 M HCl, which was mechanically cleaned with alumina, at constant potential for 10 minutes. Cyclic voltammetric (CV) experiments were performed using Autolab 101 potentiostat/galvanostat and Nova Software. The electrochemical polymerization was carried out with a three-electrode system which consisted of homemade bare GCE, Pt wire and Ag/AgCl (sat. KCl) as working, counter and reference electrodes, respectively. Electrochemical polymerization of phenol red and aniline in 0.05 mmol L^{-1} $HClO_4$ solution was carried out by CV for 25 cycles between -0.5 V to 2.0 V (vs. Ag/AgCl) with 0.05 V s^{-1} scan rate. In order to obtain a polymer-coated nanocomposite surface, aniline and phenol red solutions using different ratios (5-10-25-50%) of monomer solutions were prepared.

2.3. CV, SEM and XPS instrumentation

Cyclic voltammetric (CV) measurements were carried out utilizing an Autolab 101 potentiostat/galvanostat and Nova Software. The electrochemical measurements were performed using a three-electrode system consisting of polyaniline and polyphenol red modified electrodes, Pt wire, and Ag/AgCl (sat KCl) as working, counter and reference electrodes, respectively.

To investigate the morphology of samples with SEM, the samples were coated with a gold (Au) layer that was few nanometers thick using the Leica EM ACE600 coating equipment (Leica Microscopes, Milton Keynes, United Kingdom). Since all the samples had previously been stored in a desiccator following the XPS studies, the typical drying step was not carried out. After being coated, the samples were analyzed using scanning electron microscopy (SEM, Thermo Scientific Apreo S LoVac SEM, Thermo Fisher Scientific, Waltham, Massachusetts, United States). The Everhart– Thornley detector (ETD) was used to record images of the scanned areas at magnifications of 5000 for each sample at 5kV.

Monochromatic Al Kα radiation (1486.7 eV) was employed in XPS (Thermo Scientific Model K-Alpha XPS - Thermo Fisher Scientific, U.K.) analysis of samples. The photoelectron binding energy scale referenced the 284.8 eV C 1s peak. Snap mode was used for each element and survey spectra scans were done at 200 eV with 1 eV step size. Analysis chamber pressure was 1 × 10^{-8} mbar. The electrodes were mounted on XPS sample holder (Ted Pella, Inc. USA) with double-sided sticky carbon tape. X-ray Spot and step sizes were 50 µm. The mapping captured 200 eV survey spectrum and 50 eV snapshot scans. Almost ~1 x 1 mm^2 area scan of the samples was made and spectra were obtained at each pixel in an array of 20 x 20 pixels. Chemical imaging was obtained by applying Principal Component Analysis (PCA) to the obtained data sets.

2.4. Chemical imaging by XPS using PCA

The Avantage Software, version 5.9925, was used to process each spectroscopic data with the aid of multivariate analysis. During XPS area analyses, both survey spectra and snapshot spectra for each element were obtained. Atomic percentages (at. %) were used to express the elemental composition of each pixel. After the data sets was quantified, principal component analysis (PCA) was performed on acquired survey-XPS spectra (~1000). Each pixel denotes the possibility of the data being processed as an XPS spectrum. The methodology that was presented by Erdogan et al., 2019; 2020; Erdogan, 2022 which was utilized for the purpose of identification. After that, an improvement was made to the overall image quality. The final images were layered on top of one another so that all the components in the sample could be seen as chemical images.

3. Results and discussion

3.1. Electropolimerization of aniline and phenol red

Fig. 2 shows multisweep CVs of electropolymerization on GCE surface in 0.05 molL^{-1} $HClO_4$ containing aniline and phenol red between -0.5 V to 2.0 V vs. Ag / AgCl (sat. KCl) at a scan rate of 0.05 V s^{-1} for 25 cycles. As seen in Fig. 2, two oxidation peaks, approximately 0.56V and 0.98V, were observed. The first one shows the transformation of PANI from the reduced leukoemaraldin (LE) state to the emeraldin (EM) state, while the second one, 0.98V, shows the transition from the

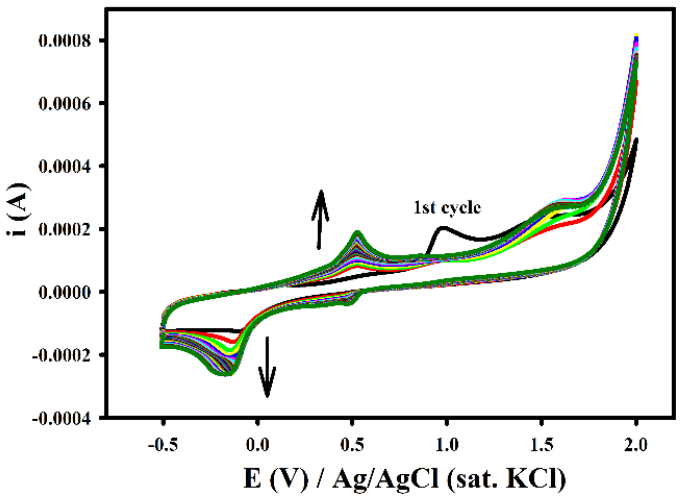

Fig. 2 Cylic voltammograms for aniline and phenol red in 0.05 molL^{-1} $HClO_4$ on bare GCE at 0.05 V s^{-1} scan rate with 25 cycles.

LE state to the pernigraniline (PE) state. It is also known that some dimers and intermediates such as p-benzoquinone, p-aminophenol are trapped in the polymer during polymerization

and the anodic bump at about 1.56V is attributed to the redox reaction of p-benzoquinone. The rise in redox currents throughout subsequent cycles indicates that the polyaniline film is growing successively (Hand and Nelson, 1974; Niu et al., 2003; Pournaghi-Azar and Habibi, 2007; Koluacik et al., 2018).

3.2. Surface analysis of polymer blends by XPS

The first information obtained from XPS is which elements are present on the surface of polymeric material. A survey, or wide-scan, spectrum over an energy range is recorded, with peaks for all elements (except H and He) in the periodic table. XPS survey spectra was collected for polyaniline and polyphenol red prior to mapping to discriminate the two polymers. In addition to survey spectra, valence band spectra are used to fingerprint polymers. Recently developed spectrometers with high counting rates and monochromators have made the study of valence bands extremely valuable for the identification of various materials. The valence-band spectrum comes from photoelectron emission from chemical bonding outer molecular orbitals. The valence band "fingerprint" energy ranges from 0 to 40 eV binding energy. Its complex band structure provides qualitative information about the chemical environment of the components that core line spectra cannot. These spectra may aid polymer identification. For this reason, both spectra were collected for two polymers. XPS survey spectra and valence band spectra for each polymer were presented in Fig.3.

According to the XPS measurements, polyaniline contains C, N and O whereas phenol red is composed of C, S and O as given in their chemical structures. Moreover, both polymers have unique valence band spectra which could be easily differentiated in area scan data processing. XPS area analyses were performed in the middle part of the electrode surface as depicted in Fig. 4. Based on the selected region, 1 x 1 mm^2 area was scanned.

The polymer blend compositions can thus be evaluated based on C, N, O and S percentages from the survey spectra, and the high-resolution C 1s, N1s and S2p photopeak shapes (data not shown). It is important to investigate the morphology of a polymer blend surface. However, the surface homogeneity can be confirmed by using XPS imaging which is also called "chemical imaging".

The use of PCA to do dimension reduction helps us simplify the entire spectrum data set by identifying the dimensions that are most prevalent within it. Following the use of PCA with the Avantage 5.9925 program in K-Alpha, one can obtain the PCA profiles (Fig. 5). PCA profiles can be designated in the form of images, as shown in Fig. 6. Photoelectron images at each individual pixel reveals the variations in concentration that occur in different locations. When PCA was applied to each polymer blend at varying concentrations, PCA1 corresponded to polyaniline and PCA2 to phenol red.

When the XPS images obtained for polyaniline mixtures are examined, although X-ray spot size is a limiting factor for XPS imaging, it has been quite successful in detecting whether polymer blends are homogeneously dispersed, especially at low concentrations.

The chemical images of polymer blends at different concentrations were designated in Fig.6. As the concentration increases the accuracy of distribution decreases due to resolution restrictions. However, with the development of new technology X-ray spot size could be decreased to 10 μm. Probably, in the future more detailed and high-resolution images will be obtained.

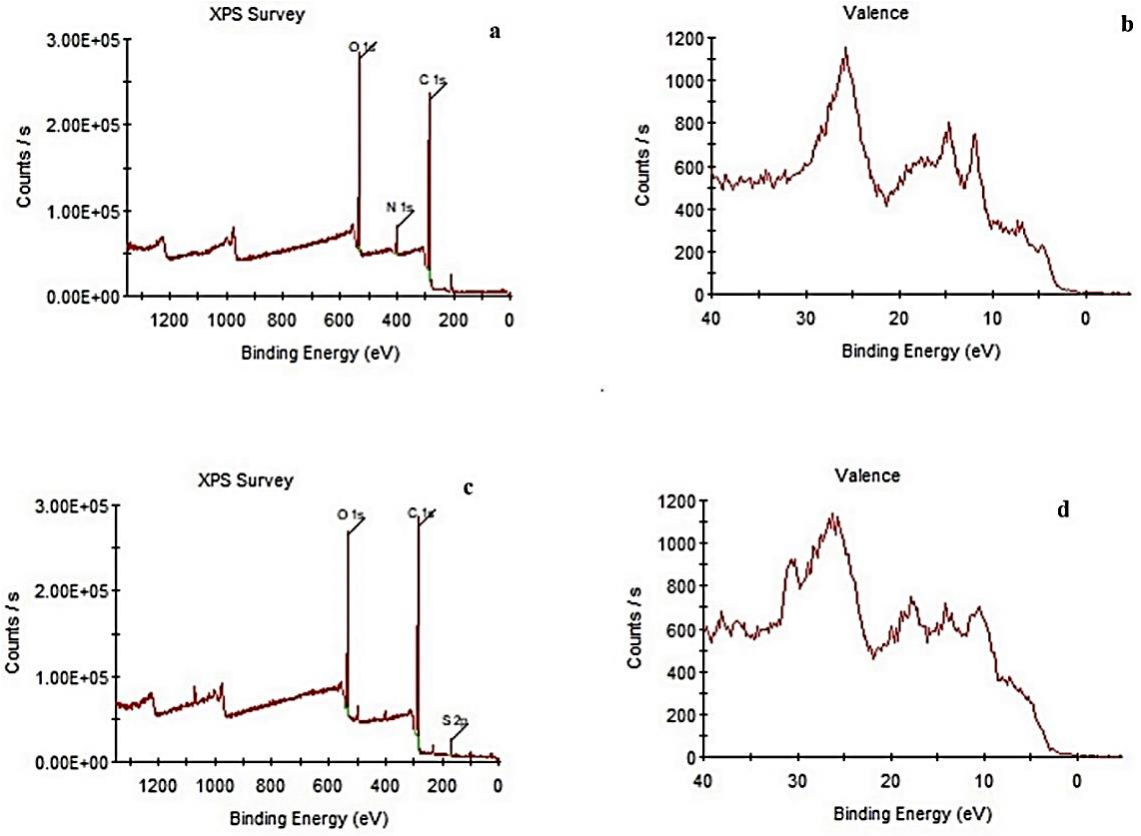

Fig. 3. XPS spectra for polymers (a, b) Survey and valence band spectra for polyaniline and (c, d) Survey and valence band spectra for polyphenol red.

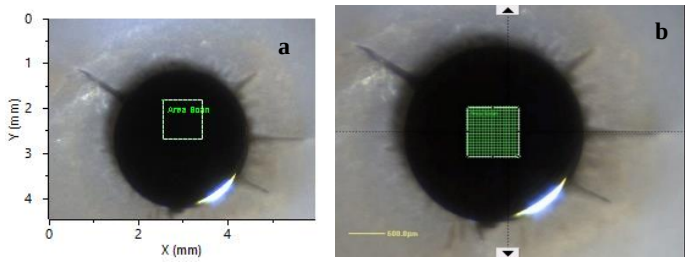

Fig. 4. Optical image of the scanned area (a) designated on a scale (b) pixel dimension (20 x 20).

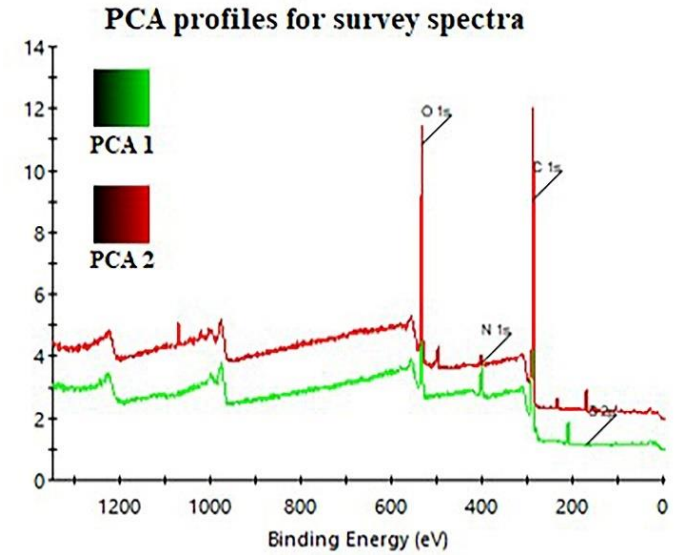

Fig. 5. XPS survey spectra of principal components for polyaniline and polyphenol red.

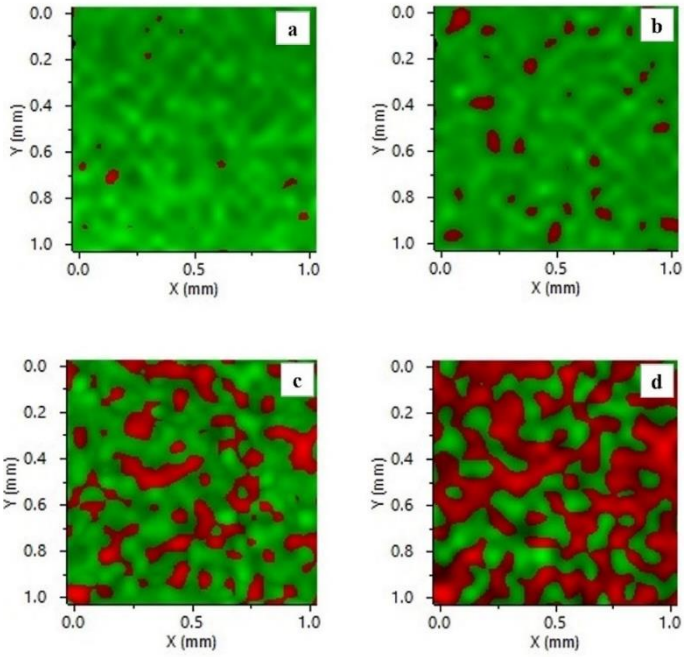

Fig. 6. XPS images of polyaniline containing phenol red (a) 5% (b) 10 % (c) 25% and (d) 50% by concentration (v/v).

3.3. Morphology of polymer blends by SEM

Due to its ease of use, specimen preparation, and image interpretation, SEM is the most popular microscopic technique. Owing to its ease of surface preparation, SEM can study all polymer structures. Surfaces of polymer blends frequently reveal important structural details and polymer distribution during preparation (Michler and Lebek, 2016). In the present study, SEM was used to give an idea about how accurate images were chemically obtained by XPS using PCA. In fact, SEM gives detailed information about the morphology of materials as well as their distribution on a small or large basis. Since XPS area scan was performed in the middle of the surfaces of the prepared samples, SEM images were also taken form the same region and with a magnification of 5000 x as presented in Fig.6. Based on SEM analyses, 5% and 10% phenol red added polymer blend areas tend to contain a significant amount of polyaniline and they are in accordance with the XPS images (Fig. 7). In addition, it is clear from these SEM images that there is phase separation occurring on the blend's surface. The data obtained from microscopy and spectroscopic imaging when combined demonstrate, as a result, that the two polymers in a mixture have separated into two distinct phases.

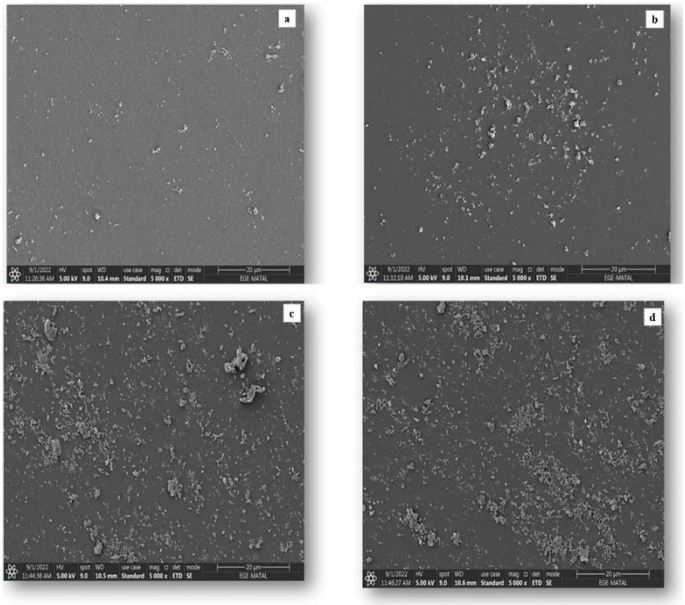

Fig. 7. SEM images of polyaniline containing phenol red (a) 5% (b) 10 % (c) 25% and (d) 50% by concentration (v/v).

4. Conclusion

In biomedical, pharmaceutical, and drug delivery applications, a single polymer often cannot perform a complex function. Blending polymers overcomes polymer limitations. Polymer blends enhance pharmaceutical and biomedical applications. Because many polymers are safe and have been used in pharmaceutical and biomedical products, many polymer blend combinations exist. To rationally select and use these polymer blends, polymer-polymer interactions must be well-understood.

This allows novel polymer blends to be designed and manufactured consistently to meet unmet needs. Pharmaceutical and biomedical applications of polymer blends and their characterization should be considered due to their localization and functionality.

In this study, chemical images of phenyl red-polyaniline mixtures prepared at different concentrations were analyzed by SEM and XPS. In addition, two different polymer mixtures were characterized by XPS and SEM. In addition, the distribution of

the polymer mixtures was determined by XPS imaging.

It should be noted that among the limitations of this technique is the X-ray spot size. This study demonstrated that XPS and SEM images acquired from the same areas on polymer blends could reveal whether the mixture is homogeneous or heterogeneous.

Both types of images were taken from the same locations on the polymer blends. The combination of the two methods results in a more comprehensive approach to the task of characterizing complex polymer blends that have distinct differences in their surface and bulk compositions.

Conflict of interest: The authors declare that they have no conflict of interests.

Informed consent: The authors declare that this manuscript did not involve human or animal participants and informed consent was not collected.

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