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Characterization and Synthesis of Acrylic Copolymer of Amidoxime

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Abstract

Following the fabrication of the copolymer membranes into DMP, the copolymer was treated with HA in an aqueous solution at temperatures ranging from 60 to 80 degrees Celsius. The Amidoximation Process alters some of the nitrile groups in the copolymer such that they are now amidoxime groups. A Number of techniques, Including "Fourier Transform Infrared, X-Ray Diffraction, and Thermogravimetric Analysis", were performed in the Characterization of Virgin and Amidoximated Acrylic Copolymer Membranes. During the amidoximation process, numerous significant changes in the properties of the copolymer, including "functionality, crystallinity, thermal behaviour, hydrophilicity, elemental composition, and surface morphology", have been observed. These changes have been attributed to a variety of different factors.

Keywords: Acrylic Copolymer, Thermal Behavior, Surface Morphology, Amidoximation, Crystallinity.

Introduction

Polyacrylonitrile, often known as PAN, has a high level of thermal stability and a high level of resistance to organic solvents. Systematically, polymers are utilised in water purification coagulation processes. Copolymerization is an essential technique for modifying the properties of economically relevant polymers. In many different types of manufacturing, acrylic esters copolymers have been found to be useful. The element PAN was utilised in the production of carbon fibres for use in clinical applications and synthetic fibres, as well as sporting and aerospace equipment.

Changing the properties of polymers may be accomplished by a wide variety of techniques, such as "grafting, plasma treatment, adsorption, blends, copolymerization of monomers, and chemical treatments, such as hydrolysis, reduction, and reaction with chemical reagents". Polymers having antimicrobial properties are either naturally antimicrobial or have been modified by the incorporation of antimicrobial agents during the production of the polymer or by the immobilisation of these biocidal agents after the creation of the polymer. It is well knowledge that nanosilver (NS) has the capacity to eradicate bacteria, fungus, protozoa, and

some viruses, even antibiotic-resistant strains of such pathogens. The dimensions (shape and size) of the particles have a significant impact on the antimicrobial efficacy of NS, with smaller particles having a stronger antimicrobial effect, according to several reports.

Materials and Methods

"Acrylonitrile (AN), Aazobisisobutyronitrile (AIBN), Toluene, Diethylether, Dimethyl formamide (DMF), Silver Nitrate (Agno3), Naoh, Hydrochloric Acid, and Polyvinyl Alcohol" are the chemical components that make up this compound (PVA) Every one of the chemicals that were utilised was of an analytical grade and was obtained from either Sigma-Aldrich or Fluka. Before being put to use, solvents went through the processes of drying and distillation as is standard. Chemicals were not purified to any further purification.

Instrumentation FTIR Spectroscopy

Using a Perkin Elmer, 1750X Fourier Transform Infrared Spectrometer, a Fourier Transform Infrared (FTIR) Analysis was Carried Out Using Potassium Bromide (Kbr) Pellets in the Resolution Range of 4000–400 Cm^{-1} at Room Temperature. Studies of X-ray diffraction were performed on the samples with the assistance of a PHILIPS, Holland, Cuka X-Ray Generator in order to track the morphological changes that occurred in the material. TGA, or thermogravimetric analysis, was performed out with the use of a Perkin Elmer STA 6000 Simultaneous Thermal. The Differential Scanning Calorimetry (DSC) Analysis of Samples Was Carried Out Using a Perkin Elmer–Pyris System at Temperatures Ranging from 50 to 300 degrees Celsius. In order to conduct an EDX analysis on virgin and approximated copolymer samples, a scanning electron microscope model called the STEREOSCAN 360 was utilised. Before undergoing analysis, the samples were covered in a gold and platinum film.

Results and Discussion

FTIR Spectroscopy of Virgin Acrylic Copolymer

Figure 1 displays the FTIR spectra of several acrylic copolymer samples with varying concentrations of acrylic acid (AA). At 15 percent AA concentration, it has been observed that the peak at 3413 Cm^{-1} gradually broadens and almost overpowers the band at 2944 Cm^{-1}, whilst the band at 2243 Cm^{-1} corresponds to the nitrile groups of AN gradually decreasing due to the decreasing nitrile moiety of polyacrylonitrile.

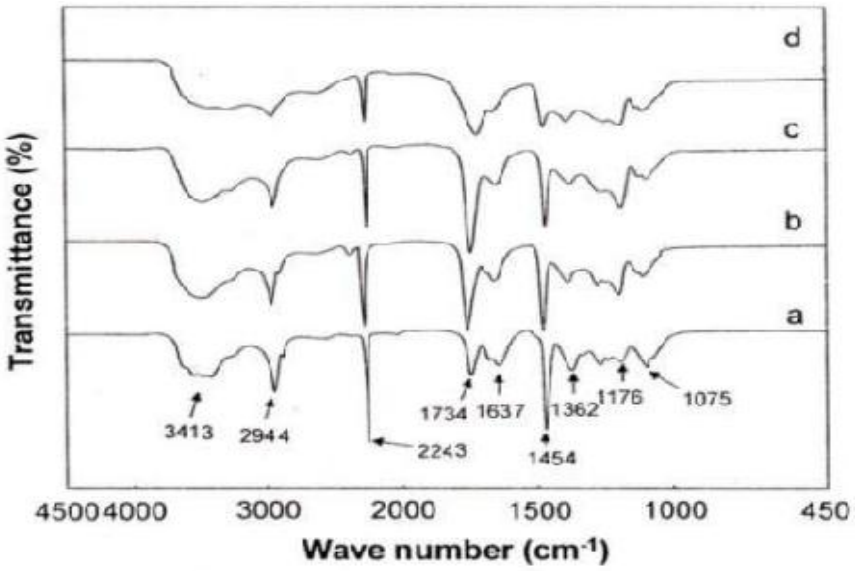

Figure 1."FTIR Spectra of Copolymer Films with Various Acrylic Acid (AA) Concentrations, (A) 2%, (B) 5%, (C) 10%, and (D) 15%"

It has been determined, based on the ratio of the peak at 1734 Cm^{-1} for carbonyl to the peak at 2243 Cm^{-1} for nitrile, how far along the process of copolymerization we are, as well as how long it will continue to take.

X-RAY Diffraction of Virgin Acrylic Copolymer

The X-Ray Analysis of the PANAA Copolymer with Various Concentrations of AA is Depicted in "Figure 2" It is possible to see that the crystalline structure dissipates and transforms into an amorphous order when the concentration of AA in the copolymer increases. It should come as no surprise that in nature, copolymers that contain 15% AA almost completely lose their crystalline structure.

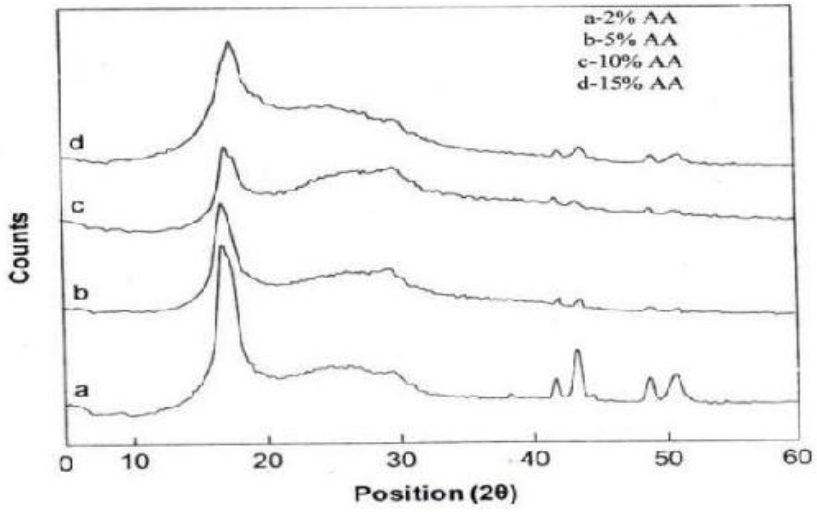

Figure 2.XRD Diffractogram of Copolymer Films with Different Acrylic Acid Concentration

$$\text{Crystallinity (percent)} = \frac{W_C}{W_O} \times 100 \rightarrow 1$$

Where W_c and W_o denote, respectively, the "weight of the crystalline region" and the "total weight of the sample".

Analysis of Amidoximated Copolymer Membranes FTIR

Figure 3 demonstrates a Decrease in the Nitrile Group Fraction That Occurs During the Amidoximation Process. Another positive formation is the appearance of a new band that is associated with the Amidoxime (AO) N-O Stretching Variation somewhere in the vicinity of 929 Cm^{-1}. Through the process of "Weiping." a similar observation was made about the PAN fibre amidoxidation.

The stretching variation of the COO group is corresponding to the peak at 1639 cm^{-1} in virgin copolymer, which can be found in the table below. Immediately Subsequent to the Amidoximation Reaction, this Region Developed "a New Peak of the –C=N- Group of Amidoxime. The –C=N- Group of Amidoxime Increases as The Amidoxime Content Increases. This Increase is so Noticeable That it Overpowers the Carboxyl Group's –C=O Band and COO. Figure 4 Shows the Band Ratio Variation Corresponding to The CN At 2243cm-1 And CH Stretching At 2944cm-1 Intensities of Virgin and Amidoximated Acrylic Copolymers".

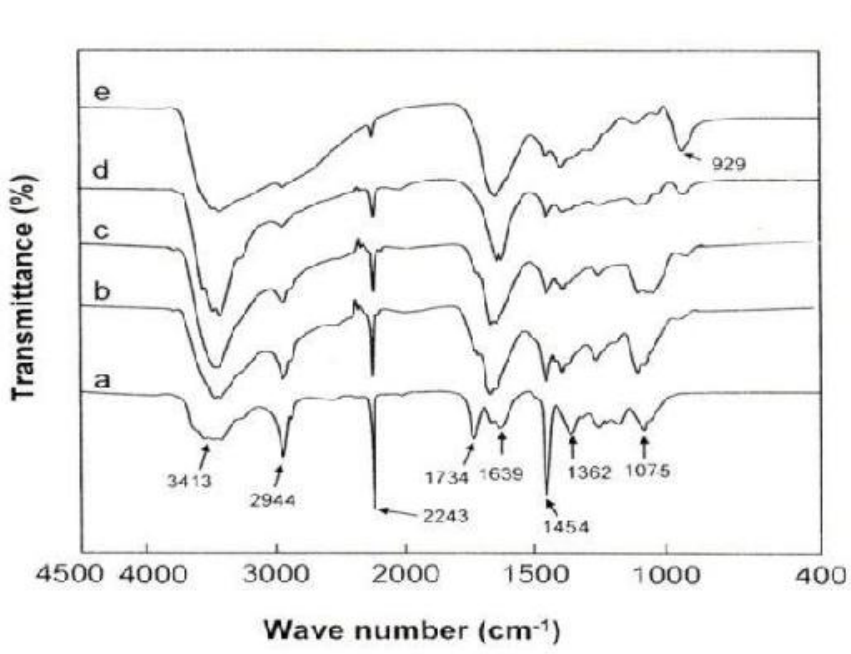

Figure 3.FTIR Spectra Of 2% Acrylic Acid Copolymer After Hydroxylamine Treatment

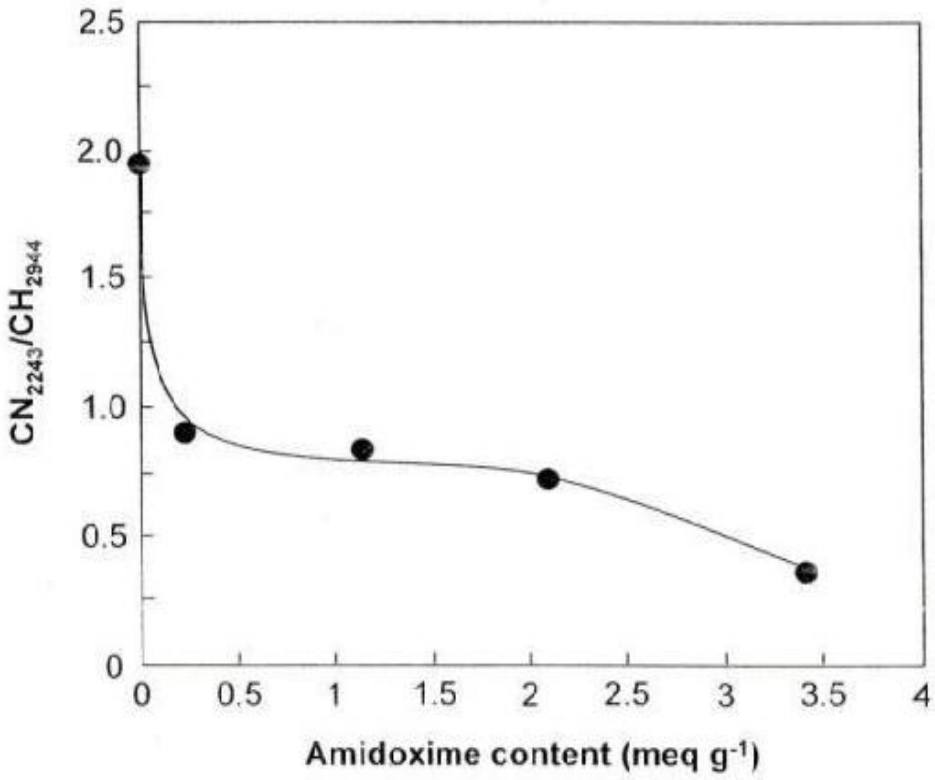

Figure 4.Variation of the Ratio of CN/CH Stretching Band with Different AO Content

This confirms that the nitrile content of the copolymer gradually decreases as a result of the conversion of nitrile groups to amidoxime groups that occurs during the amidoximation reaction, as shown by the above figure, which shows that the ratio gradually decreases as the AO content gradually increases.

X-Ray Diffraction

The XRD patterns of amidoximated copolymers and virgin copolymers are shown in Figure 5. During the amidoximation process, the Diffraction Peaks at 18^{o} and 30^{o} (2θ) become broader, which is a sign that the crystallisation shows to break apart by converting into an amorphous order. This can be seen by comparing the width of the Diffraction Peaks to the width of the Diffraction Peaks. The size of the crystal will decrease according to the amount of amidoxime present in the peak at 18 degrees.

The Crystallinity of Membranes is Examined in Relation to the Amount of AO determined in Each, and the Results are Presented in Table 1. Therefore, it is easy to observe that the crystallinity drops dramatically from 32 percent (virgin copolymer) to 14 percent (Copolymer with AO Content Of 3.3 Meq G-1).

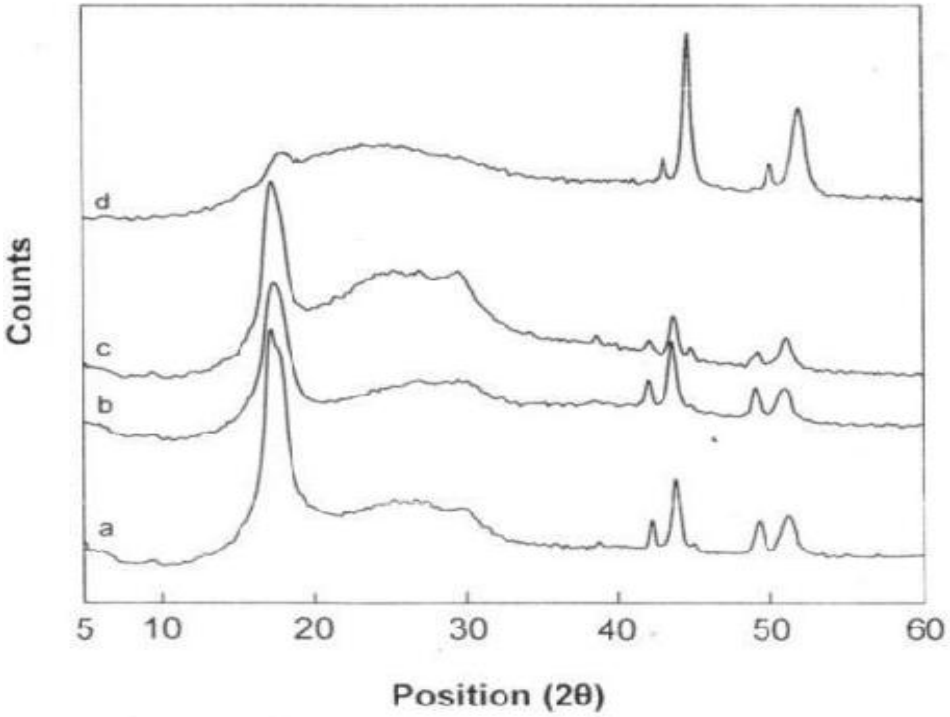

Figure 5.X- Ray Diffraction Pattern of Copolymer

TABLE 1.Percent Crystallinity of Membranes with Different AO Content

Amidoxime Content (Meq G-1)	Crystallinity (%)
Virgin Copolymer	32
0.24	23
1.12	20
3.3	14

Figure 6 is Where the Results of the Thermogravimetric Analysis (TGA) Are Presented. Up to temperatures between 270 and 300 degrees Celsius, it has been shown that pure pan does not show any loss in weight. For virgin copolymer, the degradation process takes place in three stages. In the first step, virgin copolymer degrades at temperatures ranging from 150 to 320 degrees Celsius due to the desorbed moisture, the evaporation of residual traces up to 180 degrees Celsius, and the decarboxylation of the polyacrylic portion up to 320 degrees Celsius. When PAN is heated to high temperatures, the intramolecular transformations that occur are seen to be clearly followed by intermolecular reactions of groups, which ultimately lead to cross-linking of the polymer chain. This observation was made when the PAN was heated.

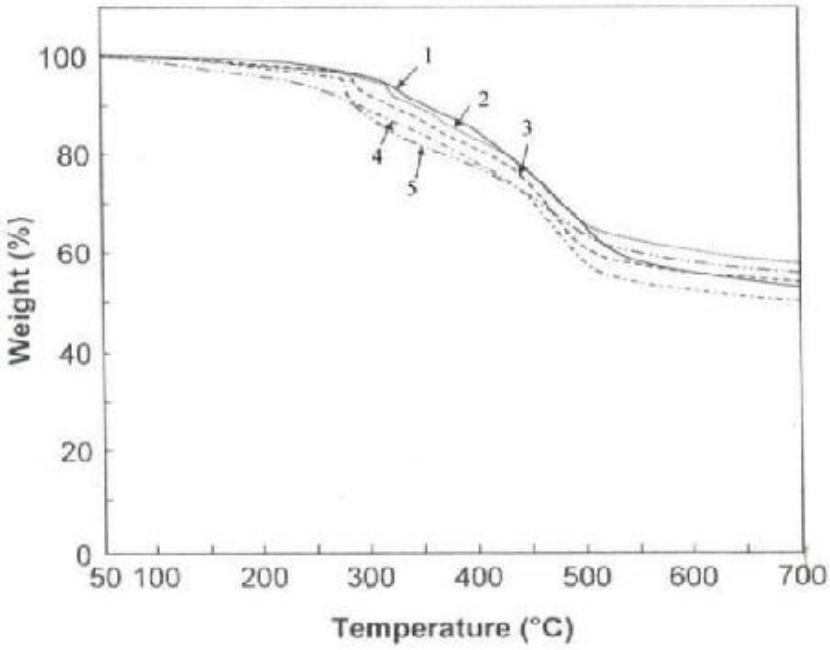

Figure 6.TGA Thermogram of Copolymer

EDX

Figure 7(A,B) is an illustration of the use of energy dispersive x-ray spectroscopy, often known as EDX, to explain the qualitative elemental composition of the virgin and oxidised samples.

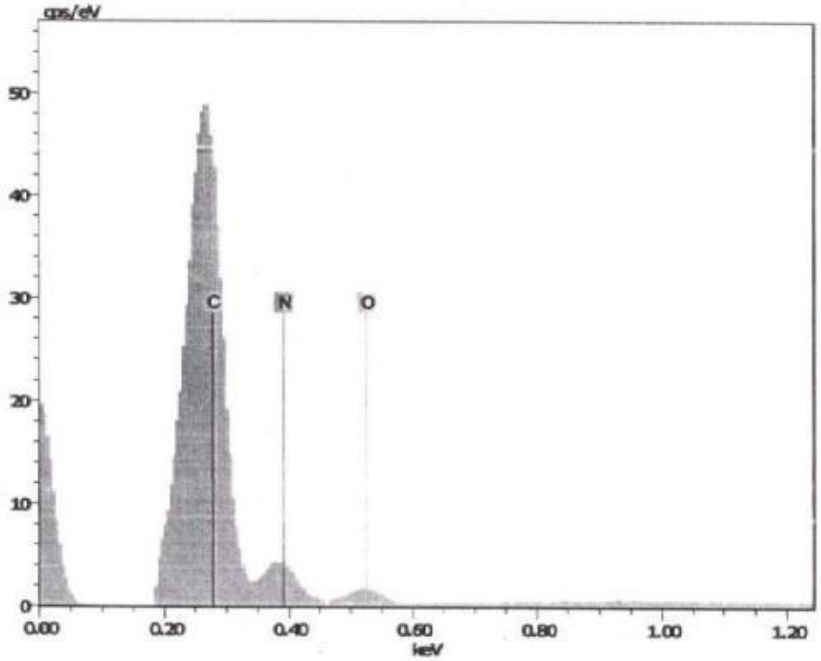

Figure 7 (A).EDX of Acrylic Copolymer Virgin and Membrane

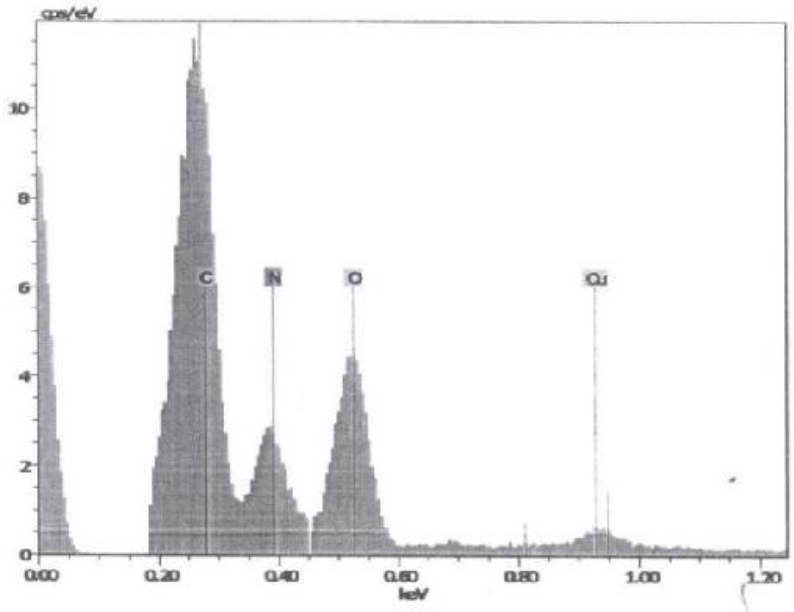

Figure 7 (B).EDX of Acrylic Copolymer AO Content 3.3 Meq/G

Figure 8 Shows the Variation of the N/C And O/C Percent Ratios with Respect to C As A Function Of AO Content.

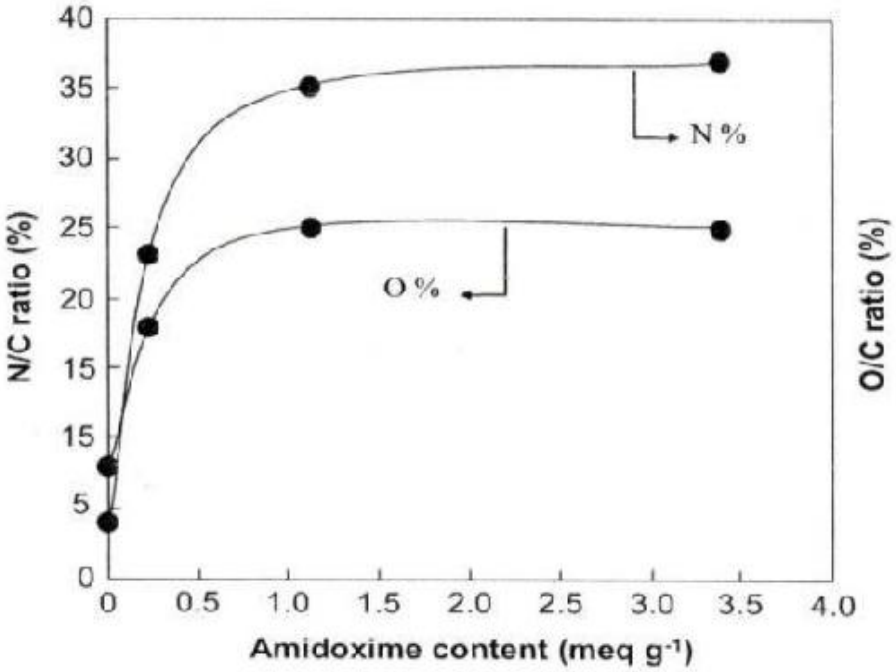

Figure 8.Determination N (%) And O (%) Content By EDX And There Variations With Respect To Carbon At Different A.O Content

Nitrogen and oxygen contents increased from approximately eight percent and four percent, respectively, to up to twenty-four percent and approximately thirty-eight percent, respectively, for virgin copolymer in samples containing three and a third milliequivalents per gramme of amidoxime. This information can be deduced from the graph that is presented in figure 8.

Conclusion

The amount of amidoxime produced is determined by the parameters of the reaction, which include the temperature, the amount of time, and the amount of HA. The FTIR study confirmed that while amidoximation continues, the nitrile content of the copolymer steadily decreases due to the conversion of nitrile groups to amidoxime groups. This was discovered as a result of the conversion of nitrile groups to amidoxime groups. In the XRD analysis, there was a correlation between the amount of amidoxime present and a decrease in crystalline diffraction. According to the results of the TGA, the initial degradation temperature of the copolymer drops as the amount of AO in the material rises. When AA is present, the temperature at which virgin copolymer begins to degrade drops significantly. The surface of the virgin copolymer will become rough as the amount of AO in the material increases, and the roughness will increase as the amount of amidoxime in the material rises.

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