

# EFFECTIVE ATOMIC NUMBERS OF POLYPYRROLE VIA TRANSMISSION METHOD IN THE ENERGY RANGE 15.74-40.93 keV

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## ABSTRACT

In the present work, effective atomic numbers ( $Z_{\text{eff}}$ ) of polypyrrole have been determined for total photon interactions at energy range 15.74 - 40.93 keV from the accurately measured total attenuation coefficients, by transmission method employing a good geometry setup for characteristic  $K\alpha$  and  $K\beta$  X-rays of Zr, Mo, Ag, In, Sb, Ba and Pr. 59.54 keV gamma rays from  $^{241}\text{Am}$  (100 mCi) source were used to excite the secondary targets. Characteristic X-rays of related elements after excited have been sent on polypyrrole using as target or absorber. Possible conclusions are compared with the theoretical ones obtained using the XCOM. The polypyrrole film has been directly formed on an aluminum plate by means of an anodization process. A Pt plate was used as the cathode. Anodization process was carried out under constant current conditions of  $I = 1\text{mA}$ . The polypyrrole/Al/polypyrrole structure was fabricated by an electrolyte being held at a constant temperature of  $55^\circ\text{C}$  that was composed of 0.40 M pyrrole and 0.1 M tetrabutylammonium tetrafluoroborate. The electrolyte solution was prepared in a propylene carbonate solvent (Merck). The polypyrrole has been coated on each side of aluminum plate. The area and thickness of the Al plate are  $1\text{ cm}^2$  and 0.012 cm, respectively.

## 1. INTRODUCTION

Studies on characteristic X-ray interaction in polymers have assumed great importance with the increasing use of these composite materials in many fields of science and technology, nuclear industry and space research programs. In such applications the concept of effective numbers ( $Z_{\text{eff}}$ ) is introduced to describe the properties of these composite materials in terms of an equivalent element. A simple and commonly employed method of obtaining  $Z_{\text{eff}}$  of a material consisting of different elements in definite proportions is based on the determination of total attenuation cross section for characteristic X-ray interaction by the transmission method and finding out an equivalent element which has the same cross section. Very few experimental results on  $Z_{\text{eff}}$  were reported for polymers for the total photon interactions at characteristic X-ray energy below 100 keV. Especially, low-energy photons are used in basic as well as applied science spanning from computerized tomography to transmission/reflection experiments for studying compound known elemental composition. All these applications of X-rays require an accurate knowledge of the probabilities of various interactions with matter. For this purpose, the available data of mass attenuation coefficients are normally used. Further at low photon energies where the photoelectric process and its associated strong  $Z$  dependence predominate. So, in the present work we have reported accurate values of  $Z_{\text{eff}}$  for polypyrrole, at energy range 15.74-40.93 keV.

We have chosen polypyrrole which has a variety of applications: electrically conducting polymers have been considered as important and interesting materials for electronic and many other applications. In the fields of molecule-based electronic device and sensor applications, it is desirable to process the conducting polymers into the thin film structure with desired thickness and molecular packing (Park et al., 2003). Among the conjugated polymers, polypyrrole (PPy) has an especially good mechanical strength, is stable in the atmosphere, and is reported to be in commercial use in batteries (Allcock and Lampe 1990). Owing to the technological importance of metal/semiconductor devices in the electronics industry (Türüt and Köleli 1993, Köleli et al., 1994, Onganer et al., 1996, Rhoederic 1998, Sze 1981) contact properties of polymers (Türüt and Köleli 1993, Köleli et al., 1994, Onganer et al., 1996), such as metallic polymer/inorganic semiconductor (Köleli et al., 1994, Onganer et al., 1996) and metal/semiconductor polymer contacts (Onganer et al., 1996, Rhoederic 1998, Sze 1981) have been extensively studied experimentally and theoretically.

## 2. EXPERIMENTAL DETAILS

The schematic arrangement of the experimental setup used in the present work is shown at İçelli and Erzeneoğlu, 2004. In the present work, values of  $Z_{\text{eff}}$  for the total photon interactions were determined from the accurately measured mass attenuation coefficient ( $\mu/\rho$ ) by transmission method employing a good

geometry setup. 59.54 keV gamma rays from  $^{241}\text{Am}$  (100 mCi) source were used to excite the secondary targets including Zr, Mo, Ag, In, Sb, Ba and Pr pure elements producing  $K\alpha$  and  $K\beta$  X-ray emission.. Characteristic X-rays of related elements after excited have been sent on polypyrrole using as target or absorber. The primer source has been housed at the center of a cylindrical lead shield of 1.2 cm diameter and 1.9 cm length. The hole diameter of source-collimator is 0.2 cm. The  $K\alpha$  and  $K\beta$  X-rays produced from secondary target have been detected through high-resolution Si(Li) semi- conductor detector having 4 mm in active diameter and 3 mm in sensitive crystal depth (FWHM of 160 eV at 5.9 keV). This detector was coupled to a computerized one connected to an ND 66 1024 multichannel analyzer through a spectroscopy automatic fine-tuning research amplifier. The weighted averages of  $K\alpha$  and  $K\beta$  energies in keV of secondary targets are given in Table 1.

**Table 1.**  $K\alpha$  and  $K\beta$  weighted averages energies of secondary targets.

| Element         | ZrK<br>$\alpha$ | ZrK<br>$\beta$ | MoK<br>$\alpha$ | Mo<br>K $\beta$ | AgK<br>$\alpha$ | AgK<br>$\beta$ | InK<br>$\alpha$ | InK<br>$\beta$ | SbK<br>$\alpha$ | SbK<br>$\beta$ | BaK<br>$\alpha$ | BaK<br>$\beta$ | PrK<br>$\alpha$ | PrK<br>$\beta$ |
|-----------------|-----------------|----------------|-----------------|-----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|
| Energy<br>(keV) | 15.7<br>6       | 17.7<br>0      | 17.4<br>4       | 19.6<br>4       | 22.1<br>0       | 25.0<br>0      | 24.1<br>3       | 27.<br>3       | 26.2<br>7       | 29.8<br>2      | 32.0<br>6       | 36.5<br>3      | 35.8<br>5       | 40.9<br>3      |

The puls height spectrum of the  $K\alpha$  and  $K\beta$  fluorescence energies by present material were collected for a period of 1800-3600 s lifetimes. The secondary target-source distance was set to 12 mm, which was determined by measuring  $K\alpha$  X-ray intensities at different distances. Secondary target-absorber distance was set to 52 mm. The counts for the measurement of each X-ray group were taken in the following sequence: no absorber ( $I_0$ ), absorber ( $I$ ). In an ideal transmission experiment, the photon once scattered in the absorber, even at very small angles, should not be detected. We assumed that in the experimental arrangement we satisfied this condition.

## 2.1. Prepare for Polypyrrole

The polypyrrole film has been directly formed on an aluminium plate by means of an anodization process. A Pt plate was used as the cathode. Anodization process was carried out under constant current conditions of  $I = 1\text{mA}$ . The polypyrrole/Al/polypyrrole structure was fabricated by an electrolyte being held at a constant temperature of  $55^\circ\text{C}$  that was composed of 0.40 M pyrrole and 0.1 M tetrabutylammonium tetrafluoroborate. The electrolyte solution was prepared in a propylene carbonate solvent (Merck). The polypyrrole has been coated on each side of aluminium plate. The area and thickness of the Al plate are  $1\text{ cm}^2$  and  $0.012\text{cm}$ , respectively.

The values of  $Z_{\text{eff}}$  for our polypyrrole were evaluated from the accurately determined molecular, atomic and electronic cross-sections, using the following sections (Kaur et al., 2000).

## 3. THEORETICAL BASIS AND CALCULATION

### 3.1. The total mass attenuation coefficients

The total mass attenuation coefficients,  $\mu_t$  is given as follows

$$\frac{\mu}{\rho} = \left( \frac{1}{\rho x} \right) \ln \left( \frac{I_0}{I} \right) \quad (\text{cm}^2/\text{g}) \quad (1)$$

Here  $I_0$  and  $I$  are respectively the intensity of beam before and after passing through an absorber.  $x$  and  $\rho$  are respectively the thickness and density of sample. The theoretical values were obtained from the state of art program XCOM and database Berger and Hubbell, 1987; also these databases have been software as WinXcom (Gerward, 2001). It depends on applying the mixture rule to calculate the partial and total mass attenuation coefficients for elements, mixtures and chemicals compounds at standard as well as selected energies.

### 3.2. The total molecular cross-section

Values of mass attenuation coefficients can be used to determine the total molecular cross section,  $\sigma_{t,m}$ , by the following relation

$$\sigma_{t,m} = \frac{1}{N} \left( \frac{\mu}{\rho} \right)_{comp} \sum_i (n_i A_i) \quad (2)$$

Here  $N$  is Avogadro's number,  $n_i$  is the number of atoms,  $A_i$  and  $\left(\frac{\mu}{\rho}\right)_{comp}$  are the atomic weight and total mass attenuation coefficient of the  $i$ th element in a molecule, respectively.

### 3.3. The total atomic cross-section

The total atomic cross-section  $\sigma_{t,a}$  can be easily determined from Eq. (2) as:

$$\sigma_{t,a} = \sigma_{t,m} \frac{1}{\sum_i n_i} \quad (3)$$

### 3.4. The total electronic cross-section

The total electronic cross-section  $\sigma_{t,e}$  for the individual element is expressed by the following formula:

$$\sigma_{t,e} = \frac{1}{N} \sum_i \frac{f_i A_i}{Z_i} \left( \frac{\mu}{\rho} \right)_i \quad (4)$$

Here  $f_i$  is the number of atoms of element  $i$  relative to the total number of atoms of all elements in the mixture,  $Z_i$  is atomic number of the  $i$ th element in a molecule, and  $\left(\frac{\mu}{\rho}\right)_i$  is total mass attenuation coefficient of the  $i$ th element in a molecule.

### 3.5. The effective atomic number $Z_{eff}$

The total atomic and electronic cross-sections are related to the effective ( $Z_{eff}$ ) through the following relation:

$$Z_{eff} = \frac{\sigma_{t,a}}{\sigma_{t,el}} \quad (5)$$

### 3.6. Electron densities $N_E$ (electrons/g)

In order to calculate electron density, Eq.5 is used: (Kaur et al., 2000).

$$N_E = \frac{Z_{eff}}{A_{top}} (N \cdot n_{top}) \quad (6)$$

$A_{top}$  is the total number of the atomic weights for polypyrrole.

The experimental mass attenuation coefficients ( $\text{cm}^2/\text{g}$ ) are obtained by can be determined from Eq. (1). If calculated values from Eq.(1) are replacing in Eq. (2), we obtain experimental total molecular cross-sections. These results have been used in Eq.(3) and have been obtained experimental total atomic cross-sections. If experimentally calculating total atomic cross-sections have been rated to theoretically calculating total electronic cross-sections in Eq.(4), we obtain experimentally effective atomic numbers for involved compounds. At last, with help Eq. (5), we obtain experimentally electron density (electrons/g). Since there are no earlier reports for polypyrrole, our results of  $Z_{eff}$  constitute the first measurement in choosing energy interval. However, as seen literature, Nayak et al., 2001 have been measured effective atomic number for polyboron at 59.5 keV. Besides; Holynska et al., 2000 and Wegrzynek, 2001 concentration of Ti, Cr, Fe and Ba were analysed for polycarbonate and polymer foils by energy dispersive X-ray fluorescence spectrometry, respectively.

## 4. RESULT AND DISCUSSION

Firstly, the experimental and theoretical total mass attenuation coefficients are listed in Table 2.

Standard deviation is estimated that the maximum errors in the measured values are less than % 3.16. The errors in the present measurements are mainly due to counting statistics, nonuniformity of the absorber, impurity content of the samples and scattered photons reaching the detector. These errors are attributed to the statistical errors in the  $I$  and  $I_0$  (<1%), sample thickness (<1%), sample weighing (<1%), geometric factor (<1%), source intensity (<1%) and systematic errors (<2%). Calculated value 0.0316 are multiple with experimental values, standard deviation ( $\pm$ ) may be estimated for each measurement to second column of Table 2.

**Table 2.** Measured (this study) and theoretical (from XCOM, Berger and Hubbell 1987/199, via WinXcom, Gerward et al., 2001) total mass attenuation coefficients ( $\text{cm}^2/\text{g}$ ) for Polypyrrole.

| Polypyrrole  |                   |             |
|--------------|-------------------|-------------|
| Energy (keV) | This study        | Theoretical |
| 15.74        | $1.043 \pm 0.032$ | 0.773       |
| 17.44        | $0.847 \pm 0.026$ | 0.617       |
| 17.70        | $0.751 \pm 0.024$ | 0.599       |
| 19.64        | $0.639 \pm 0.020$ | 0.489       |
| 22.10        | $0.544 \pm 0.017$ | 0.400       |
| 24.13        | $0.554 \pm 0.018$ | 0.352       |
| 25.00        | $0.483 \pm 0.015$ | 0.336       |
| 26.27        | $0.412 \pm 0.013$ | 0.316       |
| 27.35        | $0.446 \pm 0.014$ | 0.301       |
| 29.82        | $0.407 \pm 0.013$ | 0.276       |
| 32.06        | $0.356 \pm 0.011$ | 0.259       |
| 35.85        | $0.373 \pm 0.012$ | 0.238       |
| 36.53        | $0.328 \pm 0.010$ | 0.235       |
| 40.93        | $0.336 \pm 0.011$ | 0.220       |

As can be seen from Table 2 that total mass attenuation coefficients ( $\mu/\rho$ ) for present polypyrrole material decrease in low energy range of photons (15.74-40.93 keV). This is due to photoelectric effect and thus photon absorbed by the material and this leads sharp decrease in attenuation. Similar states are confirmed by authors (Farquharson et al., 1995, Singh et al, 1996, Kumar et al., 1996, Bhandal and Singh., 1996, Murty., 2001, Akkurt et al., 2004, İçelli and Erzeneoğlu, 2004). It can be concluded that the photon attenuation coefficients depends on the photon energy and materials' density is main contribution in the photon attenuation coefficients which is important having a variety of applications as mentioned in introduction section.

The experimental and theoretical molecular, atomic, electronic cross-sections, effective atomic numbers and electron densities are listed in Table 3-4.

**Table 3.** Theoretical molecular, atomic, electronic cross-sections, effective atomic number and density of electron of Polypyrrole

| Energy<br>(keV) | Polypyrrole             |                                 |                         |           |                        |
|-----------------|-------------------------|---------------------------------|-------------------------|-----------|------------------------|
|                 | $\sigma_{t,m}$          | $\sigma_{t,a}$<br>(barns/atoms) | $\sigma_{t,e}$          | $Z_{eff}$ | $N_E$<br>(electrons/g) |
| 15.74           | $3.472 \times 10^{-23}$ | $3.472 \times 10^{-24}$         | $2.521 \times 10^{-24}$ | 1.377     | $3.069 \times 10^{23}$ |
| 17.44           | $2.771 \times 10^{-23}$ | $2.771 \times 10^{-24}$         | $2.004 \times 10^{-24}$ | 1.383     | $3.082 \times 10^{23}$ |
| 17.70           | $2.691 \times 10^{-23}$ | $2.691 \times 10^{-24}$         | $1.943 \times 10^{-24}$ | 1.384     | $3.086 \times 10^{23}$ |
| 19.64           | $2.196 \times 10^{-23}$ | $2.691 \times 10^{-24}$         | $1.580 \times 10^{-24}$ | 1.390     | $3.098 \times 10^{23}$ |
| 22.10           | $1.797 \times 10^{-23}$ | $1.797 \times 10^{-24}$         | $1.285 \times 10^{-24}$ | 1.398     | $3.116 \times 10^{23}$ |
| 24.13           | $1.581 \times 10^{-23}$ | $1.581 \times 10^{-24}$         | $1.124 \times 10^{-24}$ | 1.405     | $3.132 \times 10^{23}$ |
| 25.00           | $1.509 \times 10^{-23}$ | $1.509 \times 10^{-24}$         | $1.071 \times 10^{-24}$ | 1.408     | $3.138 \times 10^{23}$ |
| 26.27           | $1.419 \times 10^{-23}$ | $1.419 \times 10^{-24}$         | $1.004 \times 10^{-24}$ | 1.412     | $3.148 \times 10^{23}$ |
| 27.35           | $1.352 \times 10^{-23}$ | $1.352 \times 10^{-24}$         | $9.584 \times 10^{-25}$ | 1.410     | $3.144 \times 10^{23}$ |
| 29.82           | $1.239 \times 10^{-23}$ | $1.239 \times 10^{-24}$         | $8.732 \times 10^{-25}$ | 1.419     | $3.164 \times 10^{23}$ |
| 32.06           | $1.163 \times 10^{-23}$ | $1.163 \times 10^{-24}$         | $8.168 \times 10^{-25}$ | 1.424     | $3.174 \times 10^{23}$ |
| 35.85           | $1.069 \times 10^{-23}$ | $1.069 \times 10^{-24}$         | $7.464 \times 10^{-25}$ | 1.432     | $3.192 \times 10^{23}$ |
| 36.53           | $1.055 \times 10^{-23}$ | $1.055 \times 10^{-24}$         | $7.365 \times 10^{-25}$ | 1.433     | $3.194 \times 10^{23}$ |
| 40.93           | $9.883 \times 10^{-24}$ | $9.883 \times 10^{-25}$         | $6.873 \times 10^{-25}$ | 1.437     | $3.204 \times 10^{23}$ |

It is evident from Table 3-4 that theoretical values are, in general, increase effective atomic numbers to increasing energy for polypyrrole compound. Also, total molecular, atomic and electronic cross-sections are, in general, decrease with energy. However, this change or smooth variation could not be obtained in experimental values. That is, we have obtained in some energies interval increase and some energies interval decrease. But effective atomic number for polypyrrole material is almost found to have values in the range 1.37-1.43 for theory, and 1.73-2.24 for experimental; respectively. The number of electrons per unit mass,  $N_E$ , has been determinated from Eq.(6), and the result is shown in the last column of Table 3-4. It is seen that the value of  $N_E$  is almost constant, in the range (3.06-3.20 for theory and 3.86-5.00 for experimental)  $\times 10^{23}$  electrons/g, varying little with energy and the material considered.

**Table 4.** Experimental molecular, atomic, cross-sections, effective atomic number and density of electron of polypyrrole

|                 |                                 | Polypyrrole            |           |                        |
|-----------------|---------------------------------|------------------------|-----------|------------------------|
| Energy<br>(keV) | $\sigma_{t,m}$<br>(barns/atoms) | $\sigma_{t,a}$         | $Z_{eff}$ | $N_E$<br>(electrons/g) |
| 15.74           | $4.685 \times 10^{23}$          | $4.685 \times 10^{24}$ | 1.858     | $4.141 \times 10^{23}$ |
| 17.44           | $3.805 \times 10^{23}$          | $3.805 \times 10^{24}$ | 1.898     | $4.232 \times 10^{23}$ |
| 17.70           | $3.373 \times 10^{23}$          | $3.373 \times 10^{24}$ | 1.736     | $3.869 \times 10^{23}$ |
| 19.64           | $2.870 \times 10^{23}$          | $2.870 \times 10^{24}$ | 1.816     | $4.048 \times 10^{23}$ |
| 22.10           | $2.443 \times 10^{23}$          | $2.443 \times 10^{24}$ | 1.901     | $4.238 \times 10^{23}$ |
| 24.13           | $2.488 \times 10^{23}$          | $2.488 \times 10^{24}$ | 2.212     | $4.930 \times 10^{23}$ |
| 25.00           | $2.169 \times 10^{23}$          | $2.169 \times 10^{24}$ | 2.024     | $4.512 \times 10^{23}$ |
| 26.27           | $1.850 \times 10^{23}$          | $1.850 \times 10^{24}$ | 1.841     | $4.104 \times 10^{23}$ |
| 27.35           | $2.003 \times 10^{23}$          | $2.003 \times 10^{24}$ | 2.090     | $4.658 \times 10^{23}$ |
| 29.82           | $1.828 \times 10^{23}$          | $2.003 \times 10^{24}$ | 2.093     | $4.466 \times 10^{23}$ |
| 32.06           | $1.599 \times 10^{23}$          | $1.599 \times 10^{24}$ | 1.957     | $4.363 \times 10^{23}$ |
| 35.85           | $1.675 \times 10^{23}$          | $1.675 \times 10^{24}$ | 2.244     | $5.003 \times 10^{23}$ |
| 36.53           | $1.473 \times 10^{23}$          | $1.473 \times 10^{24}$ | 2.001     | $5.003 \times 10^{23}$ |
| 40.93           | $1.509 \times 10^{23}$          | $1.509 \times 10^{24}$ | 2.196     | $4.894 \times 10^{23}$ |

It is evident from Table 3-4 that total  $Z_{eff}$  experimental and theoretical values are change in low-photon energy. Singh et al., 1996 have determined that total  $Z_{eff}$  is not change with energy range ( $10^0$ - $10^5$  MeV). However, Kumar et al., 1996 have reached that total  $Z_{eff}$  is change with photon energies lower from 100 keV in clay minerals. We have concluded that total  $Z_{eff}$  is vary for <100 keV. As seen Table3-4, the significant variation in  $Z_{eff}$  is because of the relative dominance of the partial photon interaction processes. This confirms that  $Z_{eff}$  depends upon number of elements and the range of atomic numbers in a compound. As far as known literature, the measurement technique is the first used for the measurement of effective atomic number of a polypyrrole material coating with Al with transmission method.

As seen in table 3-4, significant differences in which between experimental and theoretical values are observed in polypyrrole compounds. These differences obtaining between theory and experimental results numbers for polypyrrole material are attributed to mixture rule which can be interrogated effects on the atomic wavefunction of molecular bonding and chemical, molecular, crystalline, and thermal environment. These phenomena lead to the deviation of the experimental  $\mu/\rho$  value from that of the theoretical value, since the calculation of theoretical value has been done by considering the cross section for isolated atom. As a result, it is declared that mixture rule using both to calculate effective atomic number and interpret according to energy and number of atoms in a composition or molecular mass etc. is not suitable an approach for compounds. A few measurements have been made on the non-validity of the mixture rule (Lakshminarayana et al., 1986, Tan et al., 1988, Kerur et al., 1994, Sögüt et al., 2001). Further, the properties of the polymers are related to chemical nature, the distribution of chain lengths and the amount of additives such as fillers. These factors influence the polymeric properties such as hardness, chemical resistance, etc.(Raymond and Charles, 1998). These could have direct or indirect relating on the total mass attenuation coefficients of characteristic X-ray interaction, in addition to this; total molecular, atomic and electronic cross sections in polypyrrole materials. As a result, the present study contributes new experimental mass attenuation coefficients, molecular, atomic and electronic cross sections, electron density and effective atomic number data, using a Si(Li) detector, selected energy range 15.74-40.93 keV for polypyrrole material.

Also; If want to reach and compare more sensitive values, more experimental studies connected with different polymer are needed. So, we project to extend these measurements to various polymers preparing different method.

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