МРНТИ 29.19.21
УДК 543.426; 678.073
DOI: https://doi.org/10.51889/2020-1.1728-7901.31

STUDY OF THE ELEMENTAL COMPOSITION OF FILLERS OF IRRADIATED POLYMERIC COMPOSITE BY THE METHOD OF X-RAY FLUORESCENCE ANALYSIS

Baimolda D.[1], Cechak T.[2], Tlebaev K. B.[1]

[1]Abai Kazakh National Pedagogical University, Almaty, Kazakhstan
[2]Czech Technical University, Prague, Czech Republic,

Abstract

The article studies the elemental composition of the fillers (polytetrafluoroethylene and carbon nanopowder) of the polymer composite depending on various concentrations of fillers and electron irradiation and modified polytetrafluoroethylene (PTFE). The elemental composition of the fillers and their distribution over the depth of the polymer composite and modified polytetrafluoroethylene were studied by X-ray fluorescence analysis (XRFA) and confocal micro-X-ray analysis.

X-ray spectra of the polymer composite and modified polytetrafluoroethylene were obtained. It has been established that an increase in the concentration of one component and an irradiation dose leads to a weakening of the intensities of the spectra of the polymer composite and modified polytetrafluoroethylene.

Keywords: elemental composition, filler, polymer composite, electron irradiation, polytetrafluoroethylene, X-ray fluorescence analysis;

Аннотация

Д. Баймолда[1], Чехак Т.[2], К.Б. Тлебаев[1]
[1]Казахский Национальный Педагогический Университет им. Абая, Алматы, Казахстан
[2]Чешский Технический Университет, Прага, Чехия

ИССЛЕДОВАНИЕ ЭЛЕМЕНТОГО СОСТАВА НАПОЛНИТЕЛЕЙ ОБЛУЧЕННОГО ПОЛИМЕРНОГО КОМПОЗИТА МЕТОДОМ РЕНТГЕНОФЛУОРЕСЦЕНТНОГО АНАЛИЗА

В статье исследованы элементный состав наполнителей (политетрафторэтилена и нанопоршок углерода) полимерного композита в зависимости от различных концентраций наполнителей и электронного облучения и модифицированного политетрафторэтилена (ПТФЭ). Элементный состав наполнителей и их распределение по глубине полимерного композита и модифицированного политетрафторэтилена исследовали методом рентгенофлуоресцентного анализа (РФА) и конфокального микро-РФА-анализа.

Получены рентгеновские спектры полимерного композита и модифицированного политетрафторэтилена. Установлено, что увеличение концентрации одной компоненты и дозы облучения приводят к ослаблению интенсивностей спектров полимерного композита и модифицированного политетрафторэтилена.

Ключевые слова: элементный состав, наполнитель, полимерный композит, облучение электронами, политетрафторэтилен, рентгенофлуоресцентный анализ.

Аңдатпа

Д. Баймолда[1], Чехак Т.[2], К.Б. Тлебаев[1]
[1]Абай атындағы Қазақ ұлттық педагогикалық университеті, Алматы қ., Казахстан
[2]Чехия Техника Университеті, Прага қ., Чехия Республикасы

СӘУЛЕЛЕНДІРІЛГЕН ПОЛИМЕРЛІ КОМПОЗИТ ТОЛТЫРҒЫШТАРЫНЫҢ ЭЛЕМЕНТТІК ҚҰРАМЫН РЕНТГЕНФЛУОРЕСЦЕНЦИЯ ӘДІСІМЕН ЗЕРТТЕУ

Бұл мақалада полимерлі композит толтырғыштарының элементтік құрамы (политетрафторэтилен және көміртегі наноұнтағы) әр түрлі толтырғыштардың концентрациясы және электронды сәулелену жағдайы сондай-ақ модификацияланған(өзгеріске ұшыраған) политетрафлороэтиленге (PTFE) қатысты зерттелгені туралы айтылады. Толтырғыштардың элементтік құрамы және олардың полимерлі композит және модификацияланған политетрафторэтиленнің қаншалықты терең таралу жағдайы рентгенфлуоресценция әдісімен (РФА) және конфокалды микрорентген әдісімен зерттелді.

Композиттік және өзгеріске ұшыраған политетрафторооэтиленнің рентген спектрлері алынды. Бір компоненттің концентрациясы мен сәулелену мөлшерінің жоғары болуы полимерлі композит және модификацияланған политетрафлороэтилен спектрінің қарқындылығының әлсіреуіне әсер ететіні анықталды.

Түйін сөздер: элемент құрамы, толтырғыш, полимер композит, электрондық сәулелену, политетрафторэтилен, рентгенфлуоресценция зерттеу әдісі.

INTRODUCTION

Among the materials used in modern technology, one of the important places occupies materials, the feedstock for the production of which is powders of metals, non-metals and various compounds.

Porous materials, many anti-frictional, frictional, heat-resistant, instrumental compositions, materials with special electrical, magnetic, nuclear and other properties are currently being obtained by mainly using powder technologies.These materials are used in mechanical engineering, aviation, chemical, food, metallurgical and other industries.

Quality control of finished products of powder technologies, as well as control of technological processes, requires the determination of the chemical and phase composition of both the feedstock - powders of various materials and finished products, most of which are porous materials.

In this case, two types of problems can be distinguished: determining the average composition of a powder or porous material and determining the composition of individual particles or the distribution of the concentration of the main and impurity components in porous materials.

The second type of tasks should also include determining the degree of homogeneity of the powder (or porous material) in composition.

The determination of the average composition of powder materials is successfully carried out using traditional for metallurgical production of chemical and spectral methods of analysis, neutron activation and other methods.

However, these data are insufficient for both production control and the targeted development of new technologies. To determine the characteristics of individual particles and the distribution of properties by particles, the methods of local analysis and surface analysis are most adequate.

Among these methods, a special position is occupied by X-ray fluorescence analysis, the high locality of which (units and fractions of a micrometer) provides a fundamental opportunity to solve the problem of analytical control, and the hardware design of most modern micro analyzers combining the capabilities of a scanning electron microscope (SEM), and in some cases and transmission electron microscope, allow the determination of the composition of individual particles, morphological and particle size analysis.

The traditional methods of elemental analysis (atomic and emission spectrometry) are very laborious, expensive, and require the involvement of highly qualified personnel. Currently, for the elemental analysis of various materials in production conditions, X-ray fluorescence devices, including wearable are wider applied. This is due to significant achievements in the field of x-ray technology over the past ten years.

These devices are designed for mass consumers; the main task is the express analysis of samples of rock samples, widespread alloys, and technological control of products during production, etc.

The X-ray fluorescence method has no competitors in the analysis of waste, as it allows screening and quantification of all elements present in samples at significant (> 0.001%) concentrations, and elemental analysis is quick and cheap. The method is based on the fact that atoms of chemical elements emit characteristic radiation when excited.

The emission of characteristic spectral lines can be caused by any bombardment by accelerated particles such as electrons, photons, alpha particles and ions; or high energy radiation from an x-ray tube or other suitable radioactive source. In the field of creation of polymer composite materials for special purposes, the greatest relevance acquired polymer composites, whose properties are largely determined by the interaction of the polymer with the filler at the interface.

That is a group of new polymer composite materials was created as a result of scientific research conducted in the educational physical-technological center of the Kazakh National Pedagogical University named after Abai.

Therefore, the regulation of adhesive interaction with the aim of improving the properties of composite materials when creating functional filled polymers is an important task and requires focused basic research, including in the field of modifying the surface of the filler, changing its structure, synthesis, phase and structure formation of metal-oligomeric fillers capable of chemical interaction with reactive groups of the polymer matrix. In the synthesis of small quantities of new materials, the ability to determine the composition of small particles plays a large role. In connection with the foregoing, the development of quantitative X-ray fluorescence analysis (XRF) of powder materials is an urgent problem.

So to obtain information about the elemental compositions of the modifiers and the matrix and their distributions over the depth of the polymer composite, studies were performed using XRF and confocal micro X-ray analysis.

MATERIALS AND METHODS

Composite materials with different concentrations of components and modified PTFE by using of XRF and confocal micro-X-ray analysis were studied[1-6].

The investigated samples were polymer composite materials made in the form of tablets with different concentrations of the components:

a) 10% - PTFE nanopowder, 30% - carbon, 60% - epoxy resin

b).20% - PTFE nanopowder, 20% - carbon, 60% - epoxy resin.

c) 30% - PTFE nanopowder, 10% - carbon, 60% - epoxy resin.

The method of obtaining polymer composite materials is given in [4].

Modified PTFE samples were obtained from PTFE nanopowder in two ways, the first by pressing and sintering at a temperature $T = 410^0$ C and the second by pre-irradiating the nanopowder in vacuum with a dose of D = 0.18 MGy, followed by pressing and sintering at temperature $T = 410^0$C.

Irradiation of composites and modified PTFE samples was carried out on an ELU-6 electron accelerator with energy of 2 MeV to integral doses of 20 and 30 kGy.

The investigated samples were analyzed using XRF method, a very useful technique for a non-destructive investigation of elemental composition of materials. The principle of XRF analysis lies in emission and detection of so called characteristic X-rays. When any object is exposed to ionizing radiation (e.g. X-rays or gamma radiation), the irradiated atoms absorb its energy and characteristic X-rays are produced. Since the energy of characteristic X-rays is directly related to the atomic number of the atom, the spectrometry of emitted characteristic X-rays enables us to determine the elements present in the investigated sample.

The investigated samples were measured using the XRF setup designed at the Department of Dosimetry and Application of Ionizing Radiation at the Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague. The device contains an X-ray tube (type M47 Newton Scientific) with Rh anode and an SDD (silicon drift) detector (type X-123 SDD from the AmptekCompany).

The measurement conditions for all samples on XRF and confocal X-ray analysis were: voltage - 30 kV, current - 10 μA, spectrum acquisition time - 120 s: axis X = E [keV] is the energy of the characteristic X-rays. Y axis = Log10 (count) logarithm decimal measured pulses.

An X-ray tube with a voltage of up to 30 kV and a high current strength allow obtaining detection limits and reproducibility/accuracy for elements throughout the periodic table, as well as reducing test time.

RESULTS AND DISCUSSION

Analysis of the X-ray spectra of the composites is given in Fig. 1 – 4. They showed that at low gamma-quanta energies, characteristic fluorescence of silver-Ag, calcium-Ca, titanium-Ti, iron-Fe, zinc-Zn and gold-Au elements is observed on the spectrum. Moreover, the characteristic peaks of iron are more intensive.

At high energies, a continuous spectrum is observed.

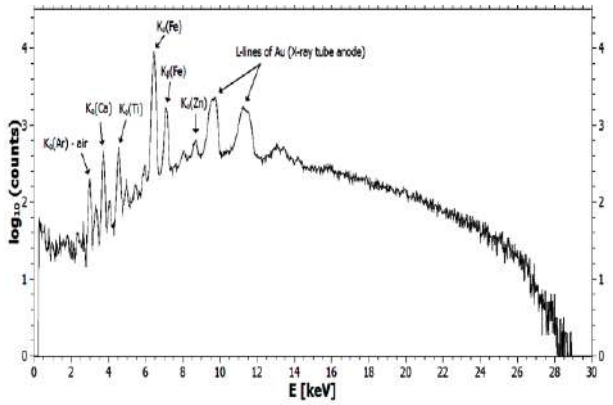


Figure 1. X-ray spectrum of the composite: 10% - Teflon, 30% - carbon, 60% - epoxy resin. Radiation dose of 30 kGy.

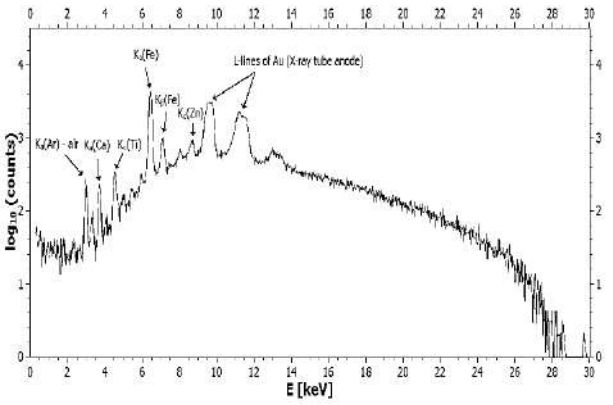


Figure 2. X-ray spectrum of the composite: 20% - Teflon, 20% - carbon, 60% - epoxy resin. Radiation dose of 20 kGy.

It was found that with increasing in the concentration of PTFE nanopowder and decreasing in the concentration of carbon in the matrix volume and with increasing in the radiation dose, the intensity of the characteristic lines is weakening, indicating a decrease in the atoms of a given substance (see Fig. 2).

The study of the distribution of the elemental composition over the depth of the sample (see Fig. 4) shows that with a low X-ray energy and with a high PTFE concentration and radiation dose, a maximum is observed, indicating the prevalence of PTFE atoms in depth.

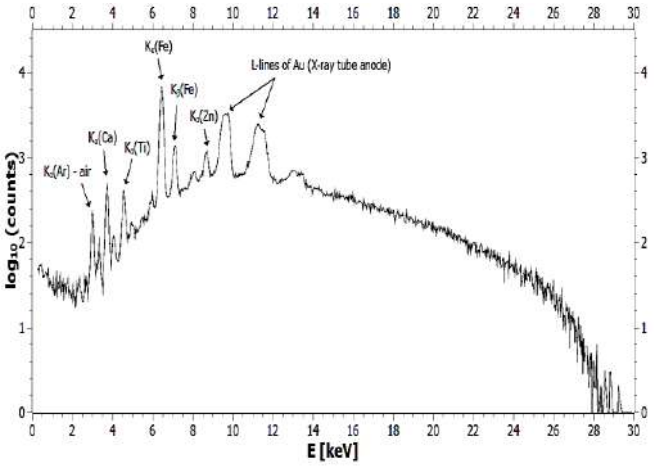


Figure 3. X-ray spectrum of the composite: 30% - Teflon, 10% - carbon, 60% - epoxy resin. Radiation dose of 30 kGy.

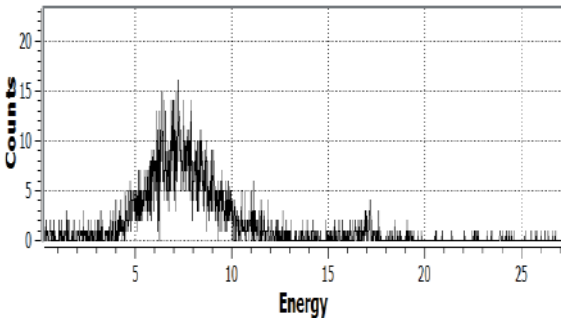
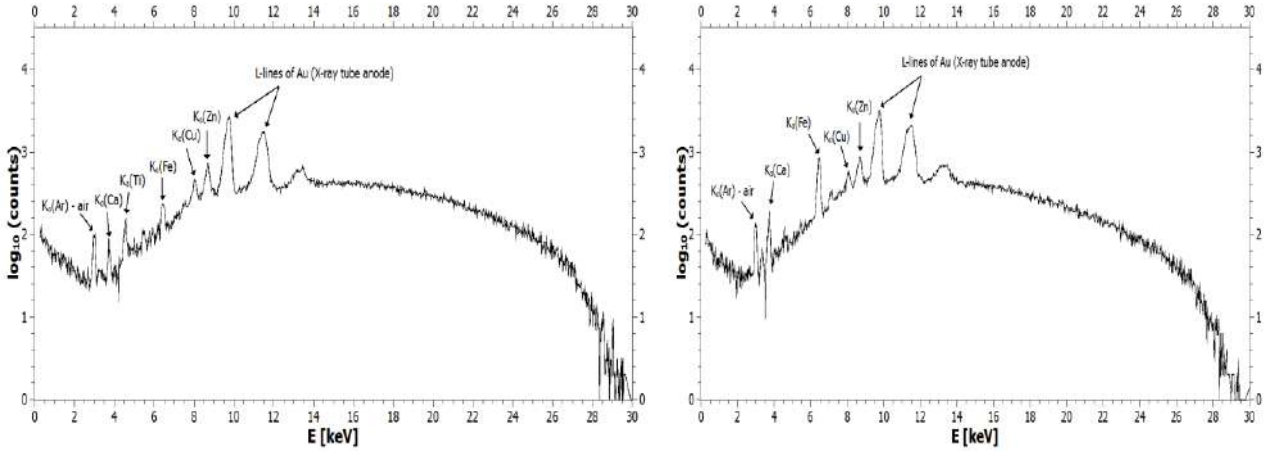


Figure 4. X-ray (3D) spectrum of the composite: 30% Teflon, 10% carbon, 60% epoxy resin. Radiation dose of 30 kGy.

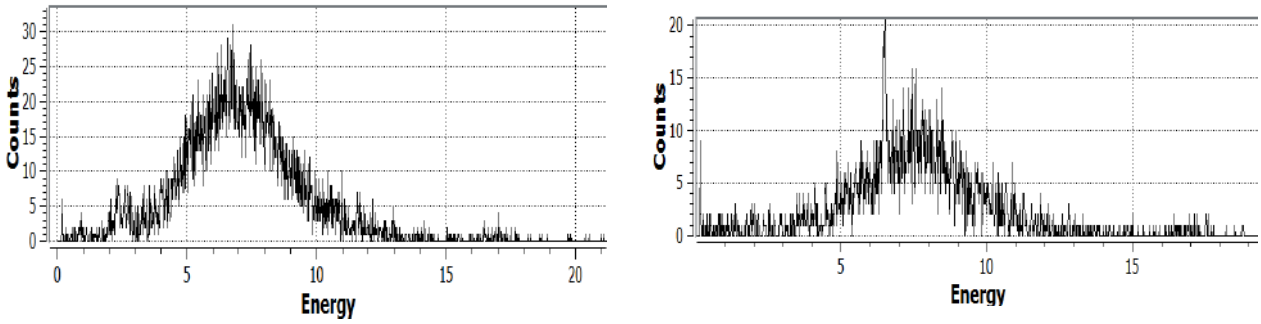
Analysis of X-ray spectra of modified PTFE samples, illustrated in Figure 5 a, b shows that the intensity of the characteristic lines of other chemical elements is significantly reduced, due to the decrease in the concentration of atoms of these substances in the sample.



a) b)

Figure 5. X-ray spectrum of modified PTFE samples obtained: a) by pressing and sintering at a temperature T = 410^0 C and b) by irradiation in vacuum with a dose of D = 0.18 MGy, pressing and sintering at a temperature T = 410^{0}C.

The data on the study of the distribution of elements in depth in the modified PTFE samples (Fig. 6 a, b) show peaks. Moreover, the peak for the first sample is more intense than for the second sample, which says about uniform, dense distributions of fluoroplastic atoms in depth.



a) b)

Figure 6. X-ray (3D) spectrum of modified PTFE samples obtained by a) pressing and sintering at a temperature T = 410^0 C and b) irradiation in vacuum with a dose of D = 0.18 MGy, pressing and sintering at a temperature T = 410^{0}C.

CONCLUSIONS

New polymer composite materials and modified polytetrafluoroethylene (PTFE) based on PTFE and carbon nanopowders have been investigated. The results of elemental analysis of new composites, performed by x-ray fluorescence spectroscopy are presented for the first time.

The research results allow making the following conclusions.

They showed that at low gamma-quanta energies, characteristic fluorescence of silver-Ag, calcium-Ca, titanium-Ti, iron-Fe, zinc-Zn and gold-Au elements is observed on the spectrum.

Moreover, the characteristic peaks of iron are more intensive. At high energies, a continuous spectrum is observed.

It was found that with increasing in the concentration of PTFE nanopowder and decreasing in the concentration of carbon in the matrix volume and with increasing in the radiation dose, the intensity of the characteristic lines is weakening, indicating a decrease in the atoms of a given substance.

The dispersed carbon filler with respect to PTFE nanoparticles is not homogeneous and consists of impurities that do not exhibit structural activity and do not affect the morphology and degree of order of the modified polymer matrix.

With an increase in the concentration of one filler in the volume of the matrix and the dose of radiation, significant attenuations of the intensity of characteristic lines are observed.

Analysis of X-ray spectra of modified PTFE samples showed that the intensity of the characteristic lines of other chemical elements is significantly reduced, due to the decrease in the concentration of atoms of these substances in the sample. The distribution of elements in depth in the modified PTFE samples exhibits peaks. It was found that the peak for the first sample is more intense than for the second sample, which says about uniform, dense distributions of fluoroplastic atoms in depth.

A significant change in the morphology and phase composition of the supramolecular structure of the polymer matrix is possible with the use of fillers with a more developed specific surface.

References:

1. Baimolda D. Application of X-ray fluorescence analysis in the coal industry// Monograph, -2018, Ulagat publishing house, Almaty.

2. Musilek L., Cechak T., Baimolda.D., Wolterbeek H. XRFA in Monitoring Impact of Coal Burning// Radiation Physical and Chemistry Journal. 1998. no. 4-6, p. 627-628

3. Baimolda.D., Cechak T.,Determination of ash content in coal by X-ray fluorescence analysis // Bulletin of KazNPU named after Abai. // 2018. -№1(61).Pages 143-147.

4. Tlebaev K.B, Kupchishin A.I. Properties of a composite with different contents of polytetrafluoroethylene nanopowder irradiated with electrons // Proceedings of higher educational institutions, № 9/2, 2016. - p. 184-189.

5. Baimolda D., Cechak T., Kulbek M., Khamraev Sh., Yerzhenbek B. Investigation of new ceramic materials obtained based on the ash of the Yekibastuz coal by X-ray fluorescence analysis, Polish Journal of Science,№24(2020), Vol1, p.39-46

6. Baimolda D., Cechak T., Yerzhenbek B.,Tlebaev K.B `Determination of sulphurand ash contents in the coal X-ray fluorescence method`//`Ponte` International Journal of Sciences and research. Italy, Florence. Issue 3, Mar 2019, Volume 75, DOI: 10.21506/j.ponte.2019.3.8