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Д.В.Сокольский атындағы «Жанармай,
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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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АО «Институт топлива, катализа и
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PROCESSING HOUSE HOLD POLYETHYLENE WASTE TO PRODUCE CARBON NANOTUBES

Abstract. The paper presents the results of processing household polyethylene waste by thermal degradation for the synthesis of carbon nanotubes by chemical vapor deposition. A feature of the work is that the decomposition of polyethylene samples and the synthesis of carbon nanotubes were carried out in one stage. The effect of temperature on the decomposition products of polyethylene wastes in the temperature range 200-550 ° C was investigated. It has been determined that even at a temperature of 450 ° C, the decomposition of polyethylene proceeds sufficiently to form carbon nanotubes. The catalyst for the growth of CNTs was cenospheres obtained from the ash and slag waste of thermal power plants during coal combustion. The main components of cenospheres are silicon and aluminum oxides. The active phase of the catalyst was iron nitrate, which is a precursor to the formation of iron clusters on the surface of cenospheres. The decomposition of polyethylene waste and the synthesis of carbon nanotubes was carried out one-stage in a tubular CVD reactor in a nitrogen atmosphere. The temperature of the synthesis of CNTs is 800 ° C. As a result of synthesis, carbon nanotubes with a diameter of 15-28 nm are formed on the surface of cenospheres, which is confirmed by results of SEM analysis and Raman spectroscopy. Based on the studies, the authors proposed a method for processing polyethylene waste for the synthesis of high quality CNTs.
Key words: carbon nanotubes, polyethylene waste, IR spectroscopy, electron microscopy.

INTRODUCTION

Currently, the problem of recycling polymer waste is a critical point in ensuring environmental safety. According to the report of the Eurasian Economic Commission, the world production of polymers in 2013 amounted to 245 megatons [1]. Polymeric waste, which makes up about 40% of all household and industrial waste, is recycled only in small quantities, and is either incinerated or disposed of in landfills. According to [2], in 2015, only 9% of the total volume of plastic waste was recycled, 12% was burnt, and 79% was collected in landfills. There are several ways to recycle plastic waste: mechanical recycling; processing of raw materials (monomerization, blast furnace recovery, chemical processing of raw materials of coke ovens, gasification, liquefaction, etc.); thermal processing (cement kilns and power generation) [3].

The review [4] presents data on the processing of polymer wastes to produce carbon nanomaterials (fullerenes, carbon nanotubes, graphenes, etc.). It is shown that the processing process is energy and resource intensive, however, with the right approach and organization of the process, it is possible to achieve economic profitability. In [5], the authors proposed a method for recycling PET plastic bottles for the production of carbon nanostructures, including fullerenes and graphene sheets. The work [6] presents the results of the synthesis of CNTs with a diameter of 30–50 nm by catalytic pyrolysis of polyethylene waste. Interest in the production of carbon nanotubes is due to the wide range of applications of these nanomaterials in various industries: energy [7], textile industry [8], as sorbent materials for wastewater treatment [9] due to the high values of the specific surface [10].

EXPERIMENTAL PART

As the initial material, household garbage (bags and containers) with the marking PET was used. Household plastic waste was pre-shredded and cleaned. Cleaning was carried out by washing the powdered sample with hot water with the addition of a surfactant. After washing, the samples were dried under normal conditions. At the end of the drying process, the crushed samples were fused without chemical degradation in a porcelain boat by heating at temperatures no higher than 130 °C, in order to obtain compact samples of polyethylene for more convenient loading into the reactor

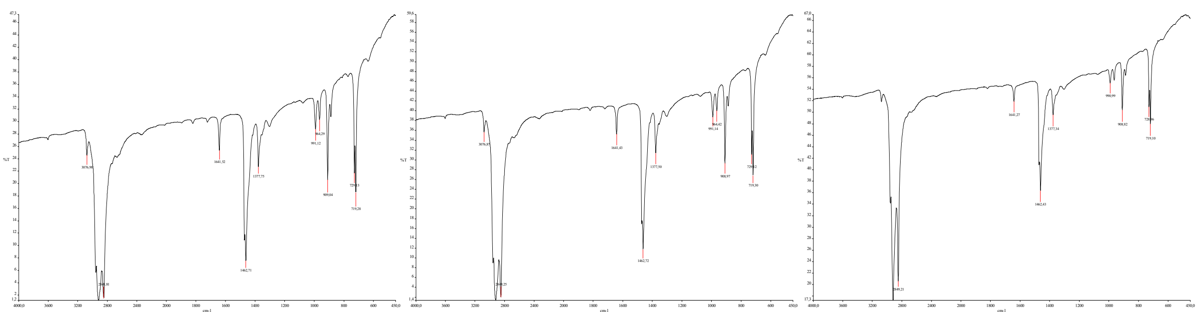
To study the decomposition process, a three-zone furnace with a quartz reactor was used. The inner diameter of the pipe is 6 cm, length 120.7 cm. A study was made of the effect of temperature on the decomposition of polyethylene. The process of decomposition of polyethylene waste in the temperature range from 200 to 550 °C was investigated. Gaseous and vaporous products of thermal degradation of polyethylene were condensed on a cellulose filter with a pore size of 2-3 μmand subjected to IR analysis on a Fourier IR spectrometer Spectrum 65.

The synthesis of carbon nanotubes was carried out by thermal destruction of polyethylene waste. The synthesis of carbon nanotubes was carried out in a three-zone CVD reactor. A quartz cuvette with polyethylene samples weighing 4 g was installed in the first zone of the reactor. $P'_{100/500}$cenosphereswere used as the basis for the catalyst for the synthesis of CNTs. To prepare the catalysts, 10 g of cenosphereswere impregnated with an aqueous solution of iron nitrate nonahydrate with a concentration of 100 g/l. Further, the catalyst samples were dried at a temperature of 70 °C for 2-3 hours until the moisture was completely removed. Quartz cuvettes with a 1 g sample of catalyst were installed in the 3rd zone of the furnace. The temperatures in the second and third zones of the furnace were set at 700 and 800 °C, respectively. Nitrogen (99.9%) with a flow rate of 530-540 cm^3/min was used as a transport gas. Synthesis time 30 min. Samples of carbon nanotubes were studied by scanning microscopy (Quanta 200i 3D B JEOL, JSM-6490LA) and Raman spectroscopy (NT-MDT NTegra Spectra), which allowed to evaluate the morphology and structure of the obtained CNT.

RESULTS AND DISCUSSIONS

It was experimentally determined that at a temperature of 200 °C thermal degradation of polyethylene waste does not occur, only a slight sintering of the samples is observed. At a temperature of 300-400 °C, a slight destruction of the sample is observed, however, this temperature is not enough for the complete destruction of polyethylene waste. Thus, it was experimentally determined that the destruction of polyethylene samples proceeds from a temperature of 450 °C. Thermal decomposition of polyethylene waste occurs with the release of white smoke with a specific odor

Figure 1 shows the IR spectrum of the thermal decomposition products of polyethylene at a temperature of 450, 500, 550 °C.



a b c

Figure 1 - IR spectra of decomposition of products of polyethylene wastes
at various temperatures: a) 450 °C;b) 500 °C; c) 550 °C

Infrared spectroscopy is one of the main methods for identifying organic materials. This is possible due to the different nature of the interaction of infrared radiation with chemical bonds and functional groups of the analyte.

Comparing the spectra presented,it should immediately be noted that all of the peaks described below are most pronounced for the pyrolysis products at 450 °C, and their presence gradually decreases with increasing temperature. So, a weak peak at a frequency of 3077 cm^{-1} corresponds to the stretching vibrations of the C-H bond present in aromatic compounds.

Peaks corresponding to a frequency of 1641 cm^{-1} characterize a double carbon – carbon bond and most likely signal the presence of alkenes in the pyrolysis products. There is other evidence of alkenes in this spectrum. So, at frequencies of 964 and 991 cm^{-1} distinct peaks were observed, corresponding to the vinyl group in trans-disubstituted alkenes and monosubstituted alkenes, respectively.

The well-structured adsorption peak at 1462 cm^{-1} seems to characterize the presence of $-CH_2$ groups.

Vibrations of C-O bonds observed at 909 cm^{-1} indicate the presence of spirits, esters and carboxylic acids. The presence of carboxylic acids is also evidenced by the band clearly pronounced in all spectra at 1377 cm^{-1}.

The bending vibrations of the C-H bonds correspond to the peaks at 729 cm^{-1} and 719 cm^{-1}, which correspond to aromatic compounds, in particular, a monosubstituted benzene ring and a phenyl group.

A group of peaks is also observing, the severity of which increases with increasing pyrolysis temperature, and with decreasing temperature the peak intensity decreases. Thus, in the range of 2915–2940 cm^{-1}, peaks corresponding to aliphatic C-H bonds are observed in all three spectra, and vibrations at a frequency of 2849 cm^{-1} correspond to symmetric methyl groups. Thus,these peaks correspond to the presence of alkanes in the samples.

Thus, the treatment of polyethylene waste at low temperatures contributes to a more noticeable presence of unsaturated compounds, alkenes, aromatics, and oxygen-containing compounds in the pyrolysis products. An increase in temperature during the pyrolysis of polyethylene in turn contributes to an increase in the formation of alkanes.

The results of IR analysis showed that there was no significant difference in the mechanism and products of the decomposition of polyethylene wastes in the temperature range 450-550 °C, thus, for the synthesis of CNTs, the decomposition temperature of polyethylene was 450 °C.

As indicated earlier, the catalyst matrix was $P'_{100/500}$cenospheres (P'-factory marking of cenospheres, 100/500 - cenosphere sizes from 100 to 500 μm) obtained by the flotation method from ash and slag waste of thermal power plants during coal combustion. Images of optical and electron microscopy of cenospheres are presented in Figure 2.

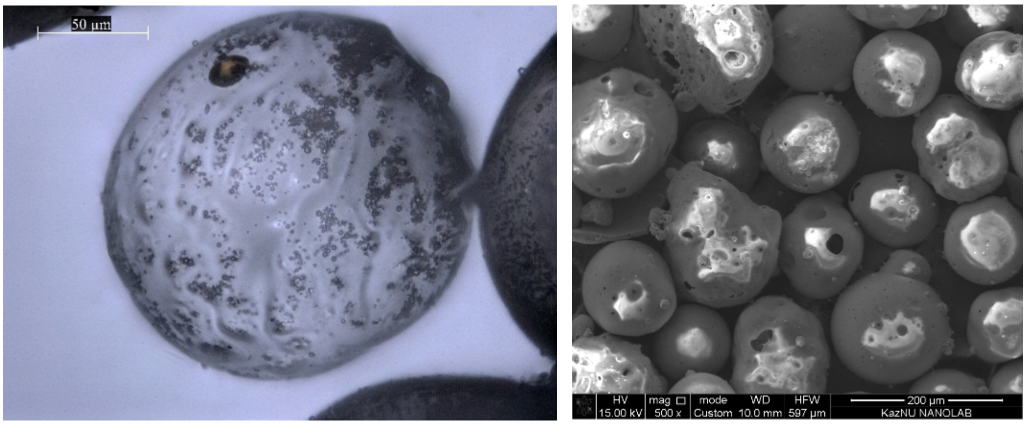
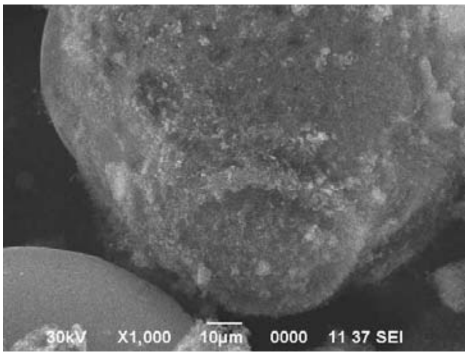


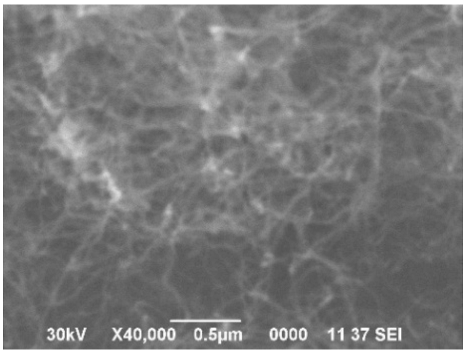
Figure 2 - Micrographs of cenospheres

The cenospheres used as the catalyst matrix are hollow spherical structures. The sizes of the spheres vary from 100 to 500 μm. The composition of the cenospheres: SiO_2 - 58-68%, Al_2O_3 - 32-38%, Fe_2O_3 - 1.4-2%, CaO - 1.9%, MgO - 1%, K_2O + Na_2O - not more than 1.5%. The melting temperature of the cenospheres is 1350-1500 °C. The wall thickness from the diameter of the microspheres is 5-10%.

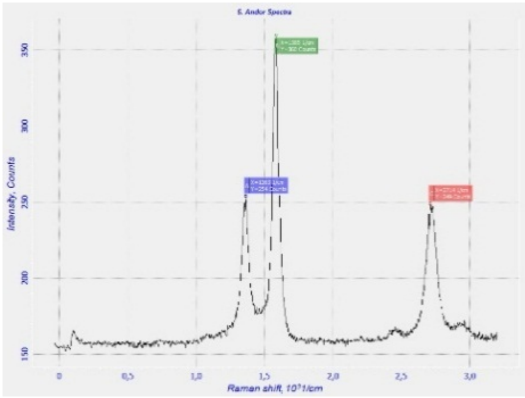
Figure 3 a,b shows SEM images of the surface of cenospheres coated with CNT.



a



b



c

Figure 3 – a, b) SEM images of the surface of cenospheres coated with CNT; c) Raman-spectra of synthesized CNTs

Analysis of SEM images of samples, obtained from polyethylene wastes at a decomposition temperature of 450 °C on a Fe@P’$_{100/500}$ catalyst, shows that in result of synthesis high-quality carbon nanotubes are forming, and completely turbostratic carbon is completely absent. Nanotubes have a diameter of 15 to 28 nm. The Raman spectrum (Fig. 3c) shows a high degree of graphitization and low defectiveness of the obtained CNTs.

CONCLUSION

Based on obtained results, a methodology for processing polyethylene wastes (PET marking) to produce carbon nanotubes is proposed. It was experimentally determined that the optimal temperature of polyethylene decomposition for the synthesis of CNTs is 450 °C. Thus, on the Fe@P'$_{100/500}$ catalyst (cenospheres with Fe $(NO_3)_2 \cdot 9H_2O$) at a synthesis temperature of 800 °C and a synthesis time of 30 min, carbon nanotubes with a diameter of 15-28 nm are formed on the surface of the cenospheres, and turbostratic carbon is completely absent. The results of Raman spectroscopy also confirm the high degree of graphitization of obtained one-dimensional carbon structures, which makes them perceptive for further practical applications.

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КӨМІРТЕК НАНОТҮТІКШЕЛЕДІ ӨНДІРУ ҮШІН ПОЛИТЕТИЛЕН ҚАЛДЫҚТАРЫН ӨҢДЕУ

Аннотация. Мақалада тұрмыстық полиэтилен қалдықтарын химиялық будың тұндыруымен көміртекті нанотүтікшелерді синтездеу үшін термиялық тозу арқылы өңдеу нәтижелері келтірілген. Жұмыстың ерекшелігі – полиэтилен үлгілерінің ыдырауы және көміртекті нанотүтікшелердің синтезі бір сатыда жүргізілуі болып саналады. 200-550 °С температура диапазонында полиэтилен қалдықтарының ыдырау өнімдеріне температураның әсері зерттелді. Тіпті 450 °С температурада да полиэтиленнің ыдырауы көміртекті нанотүтікшелерді қалыптастыру үшін жеткілікті болатындығы анықталды. КНТ өсуінің катализаторы ретінде көмірді жағу кезінде жылу электр станцияларының күл мен шлак қалдықтарынан алынған ценосфера болды. Ценосфераның негізгі компоненттері – кремний және алюминий оксидтері. Катализатордың белсенді фазасы темір нитраты болды, ол ценосфера бетінде темір кластерлерінің пайда болуының алғышарты болып табылады. Полиэтилен қалдықтарының ыдырауы және көміртекті нанотүтікшелер синтезі азот атмосферасындағы құбырлы CVD реакторында бір сатылы жүргізілді. КНТ синтезінің температурасы 800 °С құрайды. Синтез нәтижесінде диаметрі 15-28 нм болатын көміртекті нанотүтікшелер пайда болады, бұл СЭМ анализі және Раман спектроскопиясының нәтижелерімен расталады. Зерттеулер негізінде авторлар жоғары сапалы УНТ синтезі үшін полиэтилен қалдықтарын өңдеу әдісін ұсынды.

Түйін сөздер: көміртекті нанотүтікшелер, полиэтилен қалдықтары, ИК-спектроскопия, электронды микроскопия.

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ПЕРЕРАБОТКА БЫТОВЫХ ПОЛИЭТИЛЕНОВЫХ ОТХОДОВ ДЛЯ ПОЛУЧЕНИЯ УГЛЕРОДНЫХ НАНОТРУБОК

Аннотация. В статье представлены результаты переработки бытовых полиэтиленовых отходов путем термической деструкциидля синтеза углеродных нанотрубок методом химического парофазного осаждения. Особенностью работы является то, что процесс разложения полиэтиленовых образцов и синтез углеродных нанотрубок проводили в одну стадию. Было исследовано влияние температуры на продукты разложения полиэтиленовых отходов в интервале температур 200-550 °С. Установлено, что уже при температуре 450 °С разложение полиэтилена идет в достаточной степени для формирования углеродных нанотрубок. Катализаторомроста УНТ служили ценосферы, полученные из золо-шлаковых отходов ТЭЦ при сжигании угля. Основными компонентами ценосфер являются оксиды кремния и алюминия. Активной фазой катализатора служил нитрата железа, являющийся предшественником формирования кластеров железа на поверхности ценосфер. Разложение полиэтиленовых отходов и синтез углеродных нанотрубок проводили одностадийно в трубчатом CVD-реакторе в среде азота. Температура синтеза УНТ составляет 800 °С. В результате синтеза, на поверхности ценосфер формируются углеродные нанотрубки с диаметром 15-28 нм, что подтверждается результатами СЭМ-анализа и Раман-спектроскопии.На основании проведенных исследований, авторами предложен метод переработки полиэтиленовых отходов длясинтеза УНТ высокого качества.

Ключевые слова: углеродные нанотрубки, полиэтиленовые отходы, ИК-спектроскопия, электронная микроскопия

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