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NANO-grade TITANIUM DIOXIDE ON the NANOCARBON CARRIER AS CATALYST IN AEROBIC OXIDATION OF ALKYL AROMATIC HYDROCARBONS

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Abstract: *The nano-grade titanium dioxide set on multi-walled carbon nanotubes (nano-TIO_2@-MWCNTs) composite has been first tested as a potential catalyst in the aerobic oxidation of isopropylbenzene. It has been determined by changing active oxygen content that nano-TIO_2@-MWCNTs begins to actively decompose cumene hydroperoxide at temperatures above 100 degrees. Kinetic correlations and curves of oxygen uptake for cumene liquid-phase initiated (AIBN)oxidation in the presence of nano-TIO_2@MWCNTs are presented. Nano-TIO_2@MWCNTs was found to be efficient catalyst of the profound cumene liquid-phase aerobic oxidation at moderate temperatures, affording a number of valuable products. Nano-TIO_2@MWCNTs may be recommended as active catalyst for aerobic oxidation of alkyl aromatic hydrocarbons.*

Keywords: *nanotechnology, nano-grade titanium dioxide, multi-walled carbon nanotubes, liquid-phase oxidation of hydrocarbons, nanocatalysis, catalytic activity, active oxygen, reaction rate.*
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Introduction

Results of researches in the field of nanotechnology over recent years have found broad practical application, in particular, in the field of nanocatalysis. Owing to a cellular structure and a big variety of available carbon morphologies, nanotubes (CNT) are considered as promising catalysts for a number of chemical reactions. In particular, CNT are applied in industrially important gas-phase (H_2S, methane, CO, CH_4, etc.) and liquid-phase oxidation reactions (isopropyl toluene, phenol, ethanol)), as well as in a hydrogenation of CO, in the reactions of steam and carbon dioxide conversion of a methane, cycloaddition, epoxidation, cross-coupling and photocatalytic and electrochemical reactions. In the above-mentioned reactions of CNT act as carriers of the active phase of catalysts where metallics (Fe, Ni, Co, Pd, Pt, Ag, Au, etc.) and/or their oxides are applied to the surface of nanotubes, or are seeped into their pores [1-6]. The catalysis with participation of carbon nanostructures is considered as a constituent strategy of "green chemistry" and is applied to catalytic transformations in a gas and liquid phase [7].

Multiwall carbon nanotubes (MCNT), metalliferous actively catalyze aerobic oxidation of cumene [8], decalin [9] and hydrocarbons as a part of oil fraction [10, 11]. In literature there are also data on catalytic liquid-phase oxidation of cumene with air oxygen in the presence of MCNT modified on a surface by nanosilver crystals [12], catalytic oxidation of phenol and aniline with the moist air in the presence of MCNT [13-15], aerobic selection liquid-phase oxidation of ethyl benzene to acetophenone where as the catalyst served CNT, filled with iron threads (Fe@CNTs) [16]. Practically, in all above-stated processes of MCNT, acting as heterogeneous catalysts can be also used up to 5-6 times without significant loss of catalytic activity.

The actual research considers the "nano-dioxide of a titanium on a nano-carbon substrate" system as the promising catalyst of liquid-phase aerobic oxidation of alkylaromatic hydrocarbons. The purpose of this scientific investigation consists in synthesis of new composite catalyst − nano-TIO_2@MCNT and in determination of catalytic activity of the synthesized catalyst in the course of aerobic oxidation of cumene. Oxidizing reaction of isopropyl toluene is widely used as model in kinetic researches and in organic synthesis and is the most suitable for clarification of a catalytic activity, allowing to transfer the obtained data to oxidation of other hydrocarbon. Cumene is also a raw material for obtaining industrially important products – hydroperoxide compound of isopropyl toluene (HPC), acetophenone, aromatic alcohols, phenol, acetone.

Experimental part

The work consisted of several stages:

1. Composite preparation − nano-titanium dioxide on a nanocarbon substrate (multiwall carbon nanotubes) − dioxide of a titanium on a substrate from multiwall carbon nanotubes (MCNT) − nano-TIO_2@MCNT.
2. Carrying out experiments on disintegration of hydroperoxide compound of isopropyl toluene (GPK) at presence dioxide of a titanium on a substrate from MCNT − nano-TIO_2@MCNT.
3. Carrying out experiments on measuring the amount of oxygen absorbed in the model reaction of cumene oxidation initiated by 2,2'-azo-bis-isobutyronitrile in the presence of titanium dioxide on a substrate from MCNT − nano-TIO_2@MCNT.
4. Aerobic oxidation of isopropyl toluene in the presence of dioxide of a titanium on a substrate from many and wall carbon nanotubes − nano-TIO_2@MCNT.

Parent compounds and their characteristics

Procedure of synthesis of CNT and analysis of their structure and composition

Synthesis of MCNT was carried out on a laboratory installation of Scientific Instruments Dresden GMBH, SCIDRE by a pyrolysis of cyclohexane (TsG) in the presence of ferrocene (FTs) as catalyst at a temperature of 900°C within 1 hour with the subsequent chemical deposition of carbon weight from a gas phase. The synthesis precursors taken in the ratio FTs/TsG = 20 mg/ml were supplied into the reaction zone in the form of aerosol (AACVD-process) [17]. The structure and composition of MCNT were analyzed through scanning submicroscopy (SEM). External diameter of nanotubes averaged 30-40 nanometers (fig. 1).

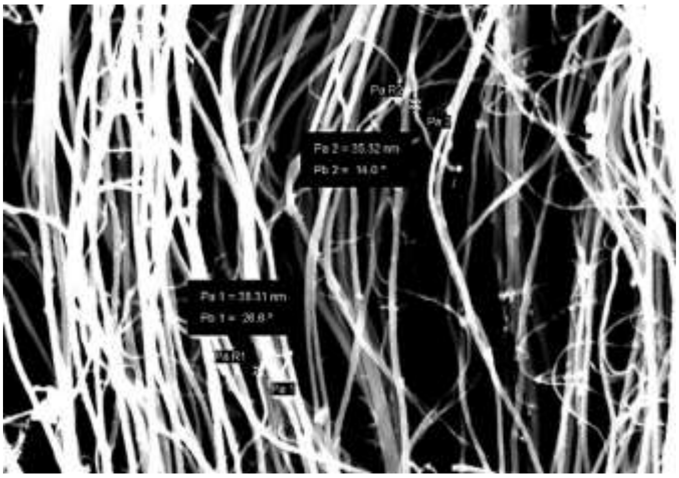
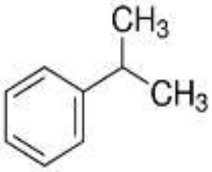


Fig. 1. A picture of a MCNT, taken by the scanning electron microscope

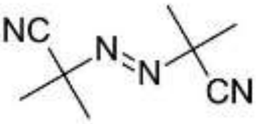
During the experiments were used:

− Titanium dioxide (TiO_2) − nano-anatase, nanoparticles size 70 nm, "ShowaDenko" (treated with hydroxyapatite), BET surface 52.1 m^2/g.

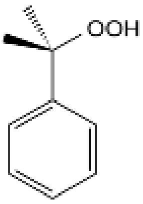
− Cumene (C_9H_{12}) - 99%, Alfa Aesar (A Johnson Mattney Company, UK). Molar mass: 120.19 g/mol. Boiling point: 152.40C. Density: 862.00 kg/m3.



− 2, 2'-azo-bis-isobutyronitrile ($C_8H_{12}N_4$, AIBN) is a colorless crystal.



− Cumene hydroperoxide ($C_9H_{12}O_2$) − 80% cumene solution, product from Merck KgaA (64271 Darmstadt, Germany). Molar mass: 152.19 g/mol. Boiling point is 153°C. Density is 1.02 g/cc.



Preparing methodology of nano-TiO_2@MCNT catalyst

The catalyst was prepared by applying TiO_2 to carbon nanotubes in a ratio of 1:10 [18]. The structure of the prepared nano-TiO_2@MCNT catalyst was analyzed by X-ray diffraction. The sample was subjected to X-ray phase analysis performed on an automatic powder diffractometer "D2 Phaser" manufactured by Bruker (Germany). The roentgenogram was taken in the following mode: CiKα-radiation, the Ni-filter, in the range of 5 ≤ 20 ≤ 75. (Bruker - D2 X-ray diffraction meter).

Fig. 2 shows a diffractogram of the catalyst. The sample consists of three phases: graphite, MCNT and anatase (nano-TiO_2).

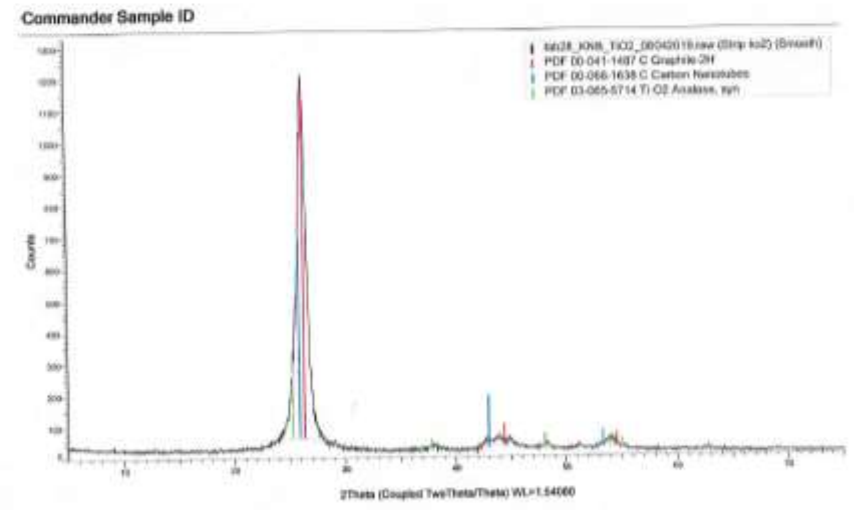


Fig. 2. XRD spectrum of the composite material nano-TiO_2@MCNT

Methodology of carrying out experiments on decomposition of cumene hydroperoxide

A 40-50 cm^3 bifurcated conical flask was used to carry out the HPC decomposition experiments which was placed in a thermostating arrangement. The active oxygen content of (ГПК) was determined by standard iodometric titration. The following reagents were used to determine the active oxygen: 50% glacial acetic acid solution (X); Soluble starch; Sodium sulphuric acid, 0.5% solution.

The breakdown of cumene hydroperoxide was carried out at different temperatures in the absence and presence of a nanocatalyst. In the course of the reaction, samples were taken (1 hour and 2 hours later) and analyzed by iodometric titration. The active oxygen content was determined by the formula:

$$A = \frac{(\mathrm{a} - \mathrm{b}) * \mathrm{K} + 0.008}{\mathrm{C}} + 100$$

Where a, b is 0.1 N of sodium thiosulfate solution used for titration of analyzed and control samples, ml;
K - correction factor 0.1 N of sodium thiosulfate solution, K = 1;
0.0008 is the amount of O_2 corresponding to 1 ml and 0.1 N of sodium thiosulfate solution;
C - sample of the test portion, g.

The reaction rate was measured by the amount of oxygen absorbed. Changes in gas volume were recorded on a gas meter unit schematically according to the procedure described in works [19,20].

The oxidation reaction of cumene with molecular oxygen was carried out on a 53 cm long oxidation unit (30 cm reverse cooler, 23 cm reactor) and 4 cm diameter. Temperature control of the oxidation column was carried out through Ultra-thermostat U1. The reaction temperature was maintained by means of a pressure thermometer (T = 120°C). Air was introduced into the reactor through the liquid reaction mixture during oxidation. Oxidation products (oxidates) were analyzed on the IR spectrum "Thermo Scientific Nicolet iS10 FTIR" and the mass spectrometer "Agilent Technologies, 7820A GS system.mm^3.

Results and discussions

It is well-known that the aerobic oxidation processes of hydrocarbons are complex chain free radical reactions consisting of a large number of parallel and sequential steps where multiple intermediates and final products are formed. Liquid phase oxidation of hydrocarbons underlies many important industrial processes, including synthetic acids and alcohols (to replace natural analogues), acetic, adipic and terephthalic acids and their esters (to produce artificial and synthetic fibre), phenol and acetone (main raw materials for plastic masses), carbonyl and ether compounds (such as solvents and special batteries), etc.

According to well-established ideas, hydroperoxides are formed as primary oxidation molecule products. Hydroperoxide molecule soders labile O−O bonds and easily undergoes further transformations to form subsequent reaction products − alcohols, ketones, aldehydes, acids, esters. Therefore, when assessing oxidation processes and their steam meters, they operate mainly on indicators related to formation and consumption of hydroperoxides.

Figure 3 shows the results of experiments to change the active oxygen content in the reaction mixture of HPK (80%) + cumene (20%) at different temperatures (Fig.3) and reaction times.

From the obtained data, it can be seen that in the absence of nano-TiO_2@MCNT additives with temperature increase, the content of active oxygen in the reaction mixture increases sharply from the initial value of 7.2% to the values of 23-34% (Fig.3A). Thus, compounds containing a peroxide O−O group are accumulated in the system and there is no appreciable consumption of these compounds even after 2 hours of reaction.

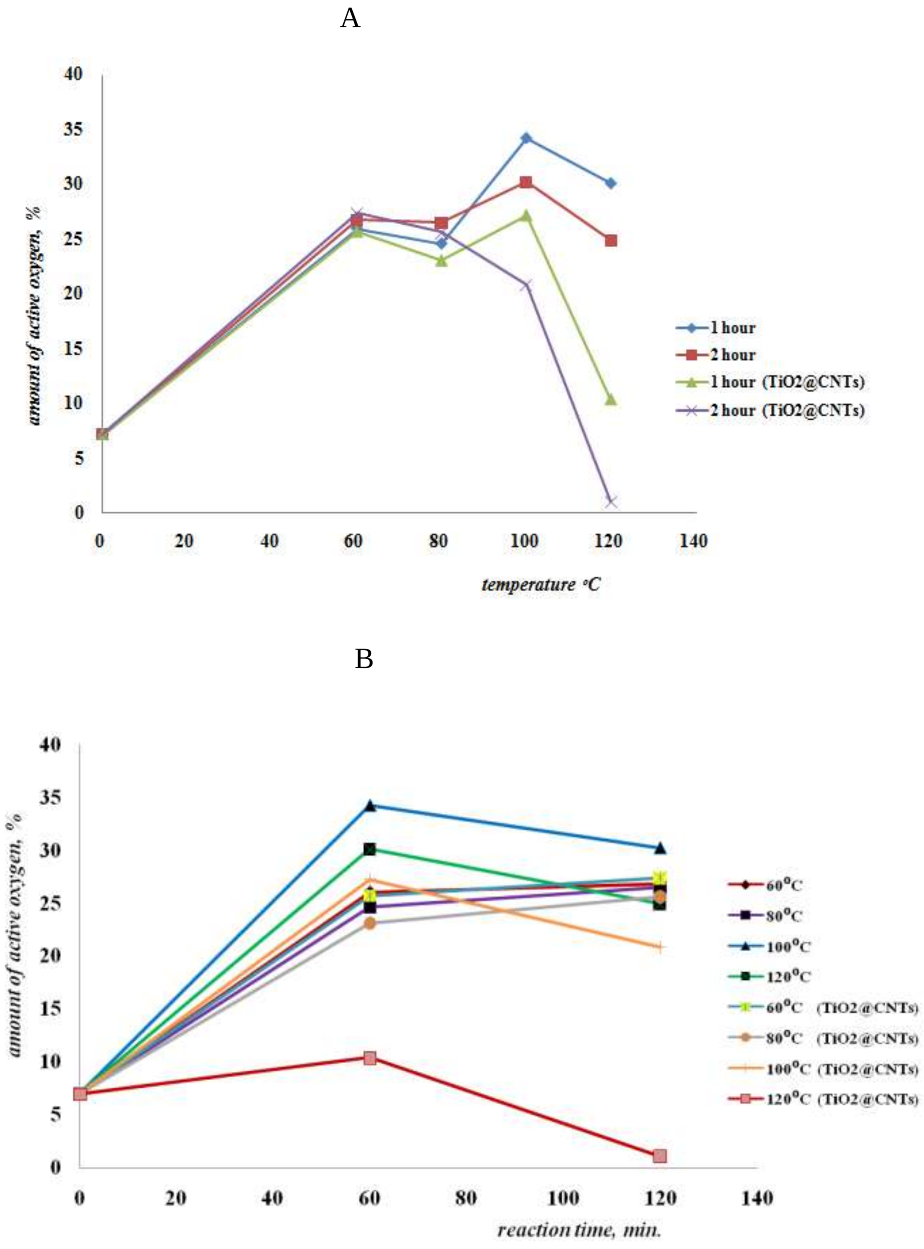
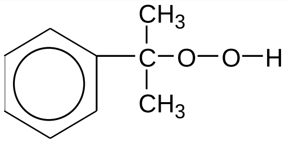
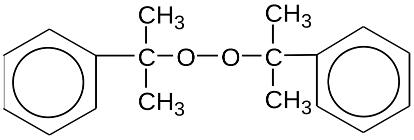


Fig. 3. Dynamics of changes in the content of active oxygen in the decomposition of cumene hydroperoxide (80% HP + 20% cumene) without catalyst (A) and in the presence of catalyst (B), at different temperatures and different reaction period.

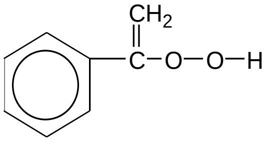
Presumably, this effect may be observed due to the additional formation of hydroperoxide by means of the cumene oxidation

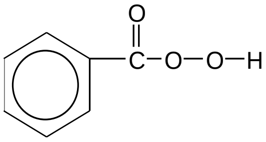


and probable formation of other peroxide compounds such as dicumyl peroxide



styrene and benzoyl hydroperoxides





In the presence of nano-TiO_2@MCNT at temperatures of 60 and 80°C, there is an increase in the active oxygen content of the system (Fig.3B). However, already at 100°C, the reaction time is 2 hours and over the whole range of reaction conditions at 120°C, the active oxygen content begins to drop sharply: at 120°C, the active oxygen content after 2 hours of reaction is only 1.05%. Thus, it can be concluded that the nano-TiO_2@MCNT compound is actively decomposing hydroperoxides formed by aerobic oxidation of cumene at temperatures above 100°C. These data are also provided by experiments to study the kinetics of oxygen uptake in the initiated oxidation of cumene.

Figure 4 shows the kinetic correlations of oxygen uptake in the presence of nano-TiO2@MCNT. The reaction was started in initiated mode (AIBN) to create a certain high concentration of GPA in the system. It can be seen from the figures that at both temperatures of 60 and 80°C the nano-TiO2@MCNT catalyst does not catalyze the active radical decay of GPC and does not contribute significantly to the radical initiation of the process – the curves are practically fused, although a small acceleration of the process, especially at 80°C, is still observed.

Thus, based on the results of experiments carried out on the breakdown of HP and the absorption of oxygen in the oxidation of cumene in the presence of nano-TiO_2@MCNT, it is possible to give certain findings on the selection of conditions for aerobic oxidation of cumene – this temperature is higher than 100°C, reaction time is not less than 2 hours, catalyst concentration is 1 wt%.

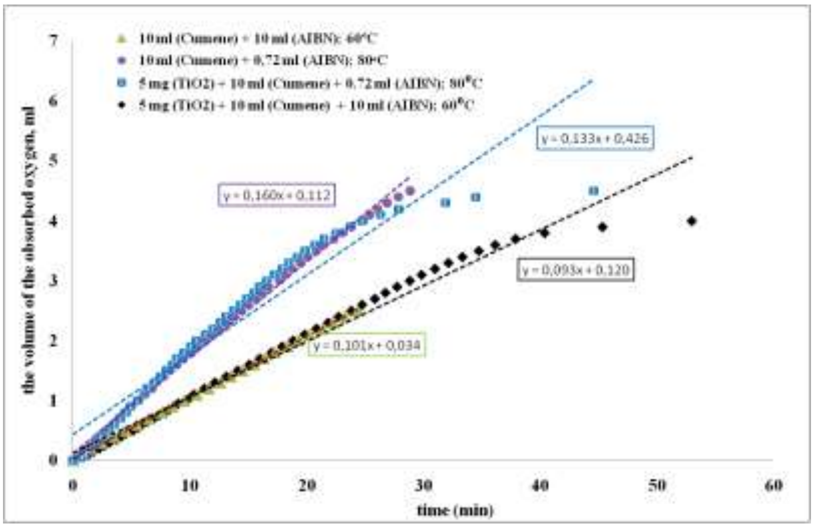


Fig. 4. Kinetic dependence of oxygen absorption in liquid phase aerobic initiated (AIBN) oxidation of cumene in the presence of nano-TiO_2@MCNT powder

As a part of the present work, during the oxidation of cumene, the volume of hydrocarbon taken was 25 cm^3, the temperature was 120°C, the reaction time was 6 hours, and the amount of nano-TiO_2@MCNT catalyst was 220 mg. The oxidate was analyzed by IC-and chromium-mass spectroscopy. Figure 5 shows the IR spectrum of the oxidate.

The following absorption bands are observed in the IR spectrum of the oxidate:

1. The band γ_{OH} = 3404 cm^{-1} due to valence fluctuations of the OH bond in the phenol molecule.
2. Bands $\gamma_{C\text{-}H}$ = 3084, 3062, 3027 cm^{-1} due to valence fluctuations of C-H bonds in arene molecule.
3. The band $\gamma_{C=O}$ = 1678 cm-1 is generated by the oscillations of the carbonyl group, this charakteric band is present near 1700 cm^{-1} in all carbonyl compounds.
4. The 1362 cm^{-1} band refers to a methyl group.
5. Bands 1602, 1583, 1449, 803 and 699cm^{-1} – characterize a monosubstituted benzene ring (fluctuations − C = C − of the molecule skeleton and C-H bonds in lateral kneading bodies).

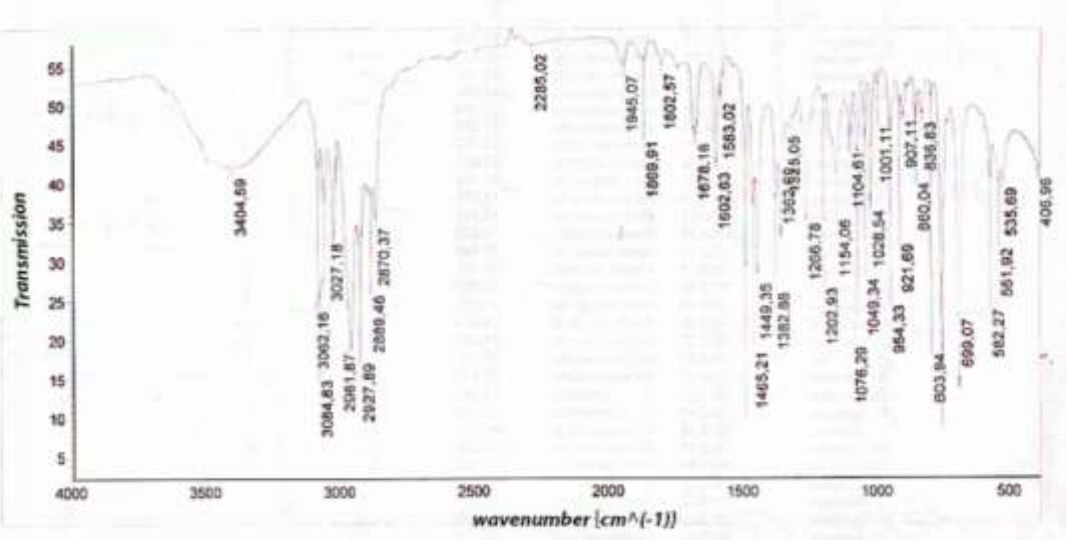


Fig. 5. IR-spectrum of products from cumene oxidation in the presence ofnano-TiO_2@ MCNT

Table provides a list of products identified by chromato-mass spectroscopy in oxidates of liquid phase aerobic catalytic and non-catalytic oxidation of cumene (data are given only for products with content exceeding 2%). From the results of the table, it can be concluded that the synthesized nano-TiO_2@MCNT composite material has a catalytic effect in the oxidation reaction of cumene with air oxygen and results in a range of oxide products, among which dicumil, cumene hydroperoxide (HP), acetophenone are isolated. A certain amount of phenol is also formed in the reaction. The obtained data provide a basis for further studies to determine optimal reaction conditions, increase hydrocarbon conversion, yield and selectivity to the desired oxidation product (Table 1).

The scheme for catalytic oxidation of cumene in the presence of nano-TiO2@MCNT can be represented as follows:

Chain initiation: $RH \xrightarrow{T:C} R\bullet$ (W_i initiation rate) (1)

Chain propagation: $R\bullet + O_2 \rightarrow RO_2\bullet$ (2)

$RO_2\bullet + RH \rightarrow ROOH + R\cdot$ (3)

Degenerate chain branching:

$ROOH + TiO_2@MCNT \rightarrow RO\bullet/RO_2\bullet + RH \rightarrow$ molecular products (4)

Chain termination

$$RO_2\bullet + RO_2\bullet \rightarrow \text{molecular products} \quad (5)$$

In the scheme, RH is a hydrocarbon, R•, RO•, ROO• is an alkyl, alkoxy and peroxide radical, respectively, ROOH is a hydroperoxide, the numbering of oxidation reactions is used and generally agreed.

Table 1. Products of the cumene oxidation (T = 120°C; t = 6 hours)

Oxidation products	**Catalyst-free, %**	**Catalyst nano-TiO$_2$@MCNT, %**
Cumene	43.846	23.622
α,α – Dicumyl	–	24.978
α – Cumene hydroperoxide	–	17.002
Acetophenone	11.561	10.32
Methylstyrene	–	6.5
2,4-Diethyl-1-methylbenzene	–	3.477
Tert-Butylbenzene	–	2.5
Phenol	–	2.28
2,4,4.6- Tetramethyl-6-phenyl-1-heptene	2.9085	–

Conclusions

1. Nano-grade titanium dioxide supported by multi-walled carbon nanotubes (nano-TiO$_2$@MCNT) composite material has been synthesized by a sol-gel method.
2. According to the results of changing active oxygen content and gas volume, it has been established that nano- TiO$_2$@MCNT actively decomposes of cumene hydroperoxide at temperatures which above 100 Celsius degrees .
3. Oxidation of cumene catalyzed by nano-TiO$_2$@MCNT was carried out in a bubble type setup at temperature 120^o C. Main products of the oxidation are hydroperoxide and acetophenone.
4. The performed study is one of the components of further results necessary for the full optimization of the process and verified recommendations for consideration in the industrial implementation.

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NANOKARBON DAŞIYICILARINDA TİTAN NANODİOKSİDLƏRİ VƏ ONLARIN ALKİL AROMATİK KARBOHİDROGENLƏRİN AEROB OKSİDLƏŞMƏSİNDƏ KATALİZATOR KİMİ TƏTBİQİ

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İlk dəfə olaraq titan nano-dioksidin nanokarbon daşıyıcısında − çoxdivarlı karbon nanoboruları (ÇKNB) substratında (nano-TiO_2@ÇKNB) izopropilbenzolun (kumol) aerob oksidləşməsi üçün potensial əlverişli katalizator kimi istifadəsi tədqiq edilmişdir. Aktiv oksigenin miqdarının dəyişməsinə əsasən müəyyən edilmişdir ki, nano-TiO_2@ÇKNB 100 dərəcədə və daha yüksək temperaturda kumolun hidroperoksidlərini aktiv şəkildə parçalayır. Maye fazada nano-TiO_2@-ÇKNB iştirakı ilə kumolun inisiator (AİBN)) iştirakında aerob oksidləşməsi zamanı oksigenin ayrılması və udulmasının kinetik asılılıq əyriləri təqdim olunmuşdur ki, bu da aktiv oksigenə əsasən alınan nəticələri təsdiqləyir. Nano-TiO_2@ÇKNB − alkilaromatik karbohidrogenlərin aerob oksidləşmə proseslərində aktiv katalizator kimi tövsiyyə edilə bilər.

Açar sözlər: nanotexnologiya, titan nanodioksidləri, çoxdivarlı karbon nanoboruları, karbohidrogenlərin maye fazada oksidləşməsi, nanokataliz, katalitik aktivlik, aktiv oksigen, reaksiya sürəti.

НАНОДИОКСИДЫ ТИТАНА НА НАНОУГЛЕРОДНЫХ НОСИТЕЛЯХ И ИХ ПРИМЕНЕНИЕ В КАЧЕСТВЕ КАТАЛИЗАТОРОВ В РЕАКЦИЯХ АЭРОБНОГО ОКИСЛЕНИЯ АЛКИЛАРОМАТИЧЕСКИХ УГЛЕВОДОРОДОВ

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Впервые исследовано применение нано-диоксида титана на подложке из многостенных углеродных нанотрубок (МУНТ)(нано-TiO_2@МУНТ) в качестве потенциального катализатора при аэробном окислении изопропилбензола (кумола). По изменению содержания активного кислорода установлено, что нано-TiO_2@МУНТ активно разлагает гидропероксид кумола при температурах 100 и выше градусов. Представлены кинетические зависимости поглощения кислорода при жидкофазном аэробном инициированном окислении кумола в присутствии нано-TiO_2@МУНТ, которые подтверждают результаты по активному кислороду. Выявлено, что нано-TiO_2@МУНТ активно катализирует реакцию аэробного окисления кумола в жидкой фазе при умеренных температурах с получением ряда ценных продуктов. Нано-TiO2@МУНТ может быть рекомендован в качестве активного катализатора аэробного окисления углеводородов.

Ключевые слова: нанотехнология, нано-диоксид титана, многостенные углеродные нанотрубки, жидкофазное окисление углеводородов, нанокатализ, каталитическая активность, активный кислород, скорость реакции.