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ТЕРМИЧЕСКОЕ РАЗЛОЖЕНИЕ ЖИДКОСТИ ДЛЯ ЭЛЕКТРОННЫХ СИГАРЕТ ПО ДАННЫМ ИК-СПЕКТРОСКОПИИ

В условиях, создающихся при использовании электронных сигарет, химические соединения, содержащиеся в них (никотин (0-2 масс. %), пропиленгликоль, глицерин, дистиллированная вода и различные ароматизаторы), могут разлагаться, и продукты разложения способны взаимодействовать друг с другом. Таким образом, могут выделяться вредные вещества и канцерогены. Встречаются эксперименты, в которых было обнаружено образование нитрозаминов, специфичных для табака, диацетилпропионила, ацетилпропионила, толуола, этилбензола, о-, м-, п-ксилола, формальдегида, ацетальдегида, ацетона, акролеина, глиоксаля.

Настоящая работа описывает исследование состава паров над жидкостью для электронных сигарет без ароматизаторов методом ИК-спектроскопии. Интервал температур составлял от 20 до 400 °C, использовался ток воздуха и ток аргона. Было установлено, что жидкость для электронных сигарет выделяет преимущественно водяной пар, диоксид углерода, пропиленгликоль и глицерин. В атмосфере аргона при температуре ~350 °C был также обнаружен акролеин. Никаких других химических соединений не было обнаружено ни в жидкой фазе методами ^{1}H, ^{13}C ЯМР-спектроскопии после нагревания до 150 °C, ни в парах при температуре ниже 250 °C.

Ключевые слова: жидкость для электронных сигарет, ИК-спектроскопия, ЯМР-спектроскопия, термическое разложение, никотин

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THERMAL DECOMPOSITION OF ELECTRONIC CIGARETTE LIQUID. IR STUDY

Under the conditions which are characteristic for electron cigarette usage chemicals containing in cigarettes (nicotine (0-2% weight usually), propylene glycol, glycerol, distilled water and different flavors) are capable to thermal decomposing and the products of decomposition could react with each other. Thus, harmful compounds and carcinogens may evolve. Literature describes a number of experiments detecting the formation of tobacco-specific nitrosamines, diacetyl and acetyl propionyl, toluene, ethyl benzene, o-, m-, p-xylene, formaldehyde, acetaldehyde, acetone, propenal, glyoxal.

The present contribution describes our efforts to study the composition of vapors over the liquids for electronic cigarettes containing no flavors by means of IR-spectroscopy. The temperature range was 20 to 400 °C, and flow of air and argon was used. The electronic cigarette liquid was found to emit aqueous vapor, carbon dioxide, propylene glycol and glycerol mostly. Propenal was also detected in vapors in argon atmosphere at temperature of ~350 °C. No other compounds were found in neither liquid phase by ^{1}H, ^{13}C NMR spectroscopy after heating to 150 ^{o}C or vapors at temperature lower than 250 °C.

Key words: electronic cigarette liquid, IR spectroscopy, NMR spectroscopy, thermal decomposition, nicotine

INTRODUCTION

Tobacco smoking is one of the most significant preventable causes of mortality in the world [1, 2]. Each year, smoking leads about 5 million people to their end [3]. The hazards of smoking are related mostly to manifold increased risks of cancer development of different king including lung [4], kidney [5], larynx [6], bladder [7], stomach cancer [8] and some others. Smoking also provides cardiovascular diseases [9, 10]. One should keep in mind that the carcinogenesis could be caused by not only tobacco combustion products but also nicotine itself [11]. Smoking harm reduction and the perspective smoking cessation are among the actual problems of modern healthcare.

Since the moment of their invention in 2004, electronic cigarette is advertised as the safe alternative for conventional cigarettes. However, to the date it is still unknown does whether electronic cigarette exclude completely all the risks intrinsic to the traditional smoking [12-15]. The liquids for electronic cigarettes (ECIG) consist of nicotine (0-2% weight usually), propylene glycol, glycerol, distilled water and, optionally, flavors. In the aerosol composition produced by ECIG, the researcher found nicotine (both free base and protonated species) [16], tobacco-specific nitrosamines (in the case if they are in ECIG liquid initially) [17], diacetyl and acetyl propionyl (by the indirect method, presumably) [18], toluene, ethyl benzene, *o*-, *m*-, *p*-xylene [19], formaldehyde, acetaldehyde, acetone, propenal [20, 21], glyoxal [22]. The data on vapors composition above ECIG liquids are not so large and detailed, and the call of F. Henkler and A. Luch «More extensive tests for e-cigarettes» outgoing from "Nature" pages [23] is fully justified.

The main aim of the present work is determining the products of ECIG liquid thermal decomposition. In order to reach this goal, the IR spectral study of vapors forming above commercially available ECIG liquid at its heating was performed. The composition of purchased ECIG liquid was confirmed employing ^{1}H and ^{13}C NMR spectroscopy.

EXPERIMENTAL

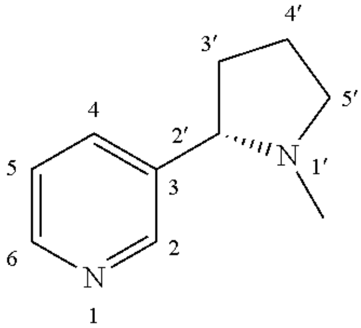
The ECIG liquid was used without preliminary preparations. Its composition claimed by manufacturer was follows: 55% weight of propylene glycol, 35% weight of glycerol, 10% weight of distilled water. Stated concentration of nicotine was 1.8 mg/ml. The liquid contained no flavors.

^{1}H and ^{13}C NMR spectra of ECIG liquid were recorded on Avance III Bruker 500 spectrometer with operating frequencies on proton of 500.17 MHz and ^{13}C of 125.77 MHz at 298.2 K temperature. The 5 mm 1H/31P/D-BBz-GRD Triple Resonance Broad Band Probe (TBI) was employed. The mixture of nitromethane, hexamethyl disyloxane and DMSO-d6 was used as external standard. Experimental error of chemical shift measurements did not exceed 0.001 ppm, the accuracy of temperature maintaining was 0.1 K using BVT-3000 thermostating unit. NMR signals identification was done employing predicted spectra [24] and the literature data [25-27].

Vibrational spectra within the range of 400-4000 cm^{-1} were registered using FT-IR spectrophotometer of Tensor 27 (Bruker Optics, Germany). The resolution of apparatus was 5 cm^{-1}, the error of wavenumber measurement did not exceed 1 cm^{-1}.

RESULTS AND DISCUSSION

The ^{13}C NMR spectrum obtained for ECIG liquid fits well enough the literature data for nicotine dissolved in D_2O at pD approximately corresponding the isoelectric point [25]. The observed deviations of chemical shifts $\delta(C_2)$ = 147.322 (150.0 [25]), $\delta(C_3)$ = 137.125 (129.9 [25]), $\delta(C_4)$ = 135.890 (138.4 [25]), $\delta(C_5)$ = 123.859 (125.9 [25]), $\delta(C_6)$ = 147.676 (151.2 [25]) are related to the differences of solvent properties. We were able to distinguish and assign the carbon signals of pyrrolidinyl group: $\delta(C_{2'})$ = 65.377 (69.2 pred. [24]), $\delta(C_{3'})$ = 33.248 (35.06 pred. [24]), $\delta(C_{4'})$ = 21.288 (22.64 pred. [24]), $\delta(C_{5'})$ = 55.902 (57.1 pred. [24]), $\delta(\underline{C}H_3)$ = 39.148 (40.46 pred. [24]).



The intensive peaks in the spectral range of 60-75 ppm should be assigned to the nuclei of carbon skeleton of polyhydric alcohols. It is possible to observe for glycerol the $\delta(\underline{C}H)$ = 71.718 (75.7 pred. [24], 73.1 [26]) and $\delta(\underline{C}H_2)$ = 62.279 (66.9 pred. [24], 63.5 [26]) signals. Two close lines of $\delta(\underline{C}H)$ = 67.305 (72.7 pred. [24], 68.2 [27]) and $\delta(\underline{C}H_2)$ = 66.504 (71.6 pred. [24], 67.8 [27]) as well as single peak in higher frequencies range of $\delta(\underline{C}H_3)$ = 18.132 (23 pred. [24], 18.6 [27]) are characteristic of propylene glycol.

In the low frequencies range of proton magnetic resonance spectrum, the signals assigned to the pyridine fragment of nicotine, $\delta(H_2)$ = 7.973 (8.528 pred. [24]), $\delta(H_4)$ = 7.363 (7.777 pred. [24]), $\delta(H_5)$ = 6.971 (7.329 pred. [24]), $\delta(H_6)$ = 7.963 (8.479 pred. [24]) could be found. Interestingly, the peaks of H_2 and H_6 atoms are located so close to each other that the H_2 singlet overlap with lower frequencies half of H_6 doublet. The signals of pyrrolydinyl group of nicotine are poorly distinguishable due to the probable overlapping with polyhydric alcohols protons in the range of 2.8-3.4 ppm, except for $-CH_3$-group (δ = 1.619, pred. 2.391 [24]). The intensive singlet at ~4.6 ppm should be referred to nitromethane of external standard. Water from ECIG liquid gives broadened peak with maximum at 4.7 ppm.

There are no other signals in the ^{1}H, ^{13}C NMR spectra. It means that the content of impurities is beyond the detection limits of the analysis method.

We should note that the ECIG liquid heated to 150 °C and then cooled to the room temperature gave the same ^{1}H and ^{13}C NMR spectra as one before being heated.

When the ECIG liquid is heated in the medium of inert gas, it is possible to distinguish 3 temperature intervals, where the gaseous products of pyrolysis evolve intensively. In the first temperature range of 40-139 °C with maximum at 102 °C, water is evaporated mostly (Fig. 1). Besides water, the significant amount of carbon dioxide could be observed in spectrum as well as little quantities of propylene glycol or glycerol. The presence of propylene glycol is more likely considering its boiling point lower by ~100 °C than that of glycerol.

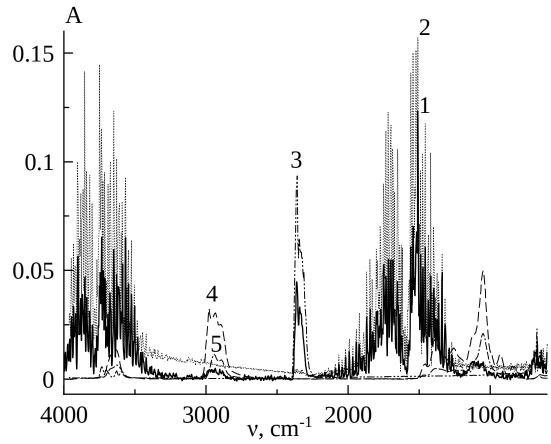


Fig. 1. IR spectrum of gaseous products of ECIG liquid pyrolysis in argon atmosphere at 102 °C: 1 – experimental, 2 – water [28], 3 – carbon dioxide [28], 4 – propylene glycol [28], 5 – glycerol [28]
Рис. 1. ИК спектр газообразных продуктов термического разложения жидкости для электронных сигарет в атмосфере аргона при 102 °C: 1 – экспериментальная кривая, 2 – вода [28], 3 – диоксид углерода [28], 4 – пропиленгликоль [28], 5 – глицерин [28]

Within temperature range of 140-300 °C, water does not evaporate anymore while the emission of carbon dioxide and polyhydric alcohols grows (Fig. 2).

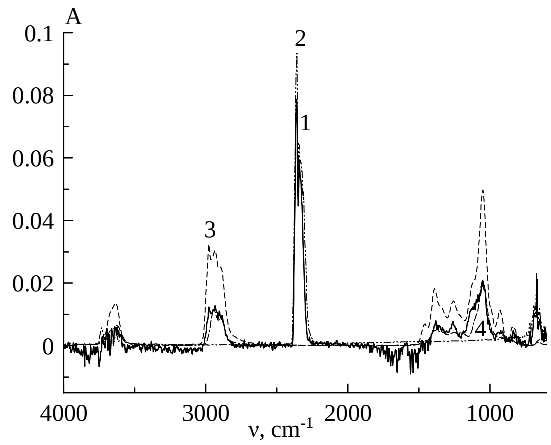


Fig. 2. IR spectrum of gaseous products of ECIG liquid pyrolysis in argon atmosphere at 178 °C: 1 – experimental, 2 – carbon dioxide [28], 3 - propylene glycol [28], 4 – glycerol [28]
Рис. 2. ИК спектр газообразных продуктов термического разложения жидкости для электронных сигарет в атмосфере аргона при 178 °C: 1 – экспериментальная кривая, 2 – диоксид углерода [28], 3 – пропиленгликоль [28], 4 – глицерин [28]

Finally, at the temperature higher than 300 °C, the transmittance lines of glycerol decomposition products, propenal and water, appear in IR spectrum of ECIG liquid vapors (Fig. 3).

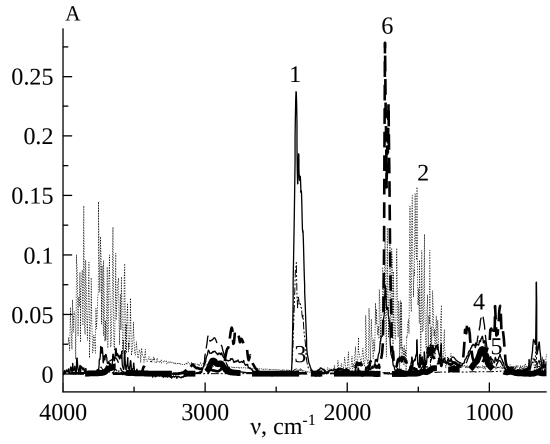


Fig. 3. IR spectrum of gaseous products of ECIG liquid pyrolysis in argon atmosphere at 348 °C: 1 – experimental, 2 – water [28], 3 – carbon dioxide [28], 4 – propylene glycol [28], 5 – glycerol [28], 6 – propenal [28]
Рис. 3. ИК спектр газообразных продуктов термического разложения жидкости для электронных сигарет в атмосфере аргона при 348 °C: 1 – экспериментальная кривая, 2 – вода [28], 3 – диоксид углерода [28], 4 – пропиленгликоль [28], 5 – глицерин [28], 6 – пропеналь [28]

Besides, the ECIG liquid probably emits some diacetyl or acetyl propionyl possessing peaks at ~1360 cm^{-1} and ~1100 cm^{-1}. Note that we were unable to detect the nicotine in IR spectra of gaseous products of ECIG liquid pyrolysis.

The heating of ECIG liquid in the air atmosphere seems to reflect the processes occurring in ECIG in way that is more realistic. The pyrolysis products evolving proceeds in two stages. In the temperature range of 45-278 °C with maximum at 216 °C, the liquid emits mostly the aqueous vapor and carbon dioxide (Fig. 4).

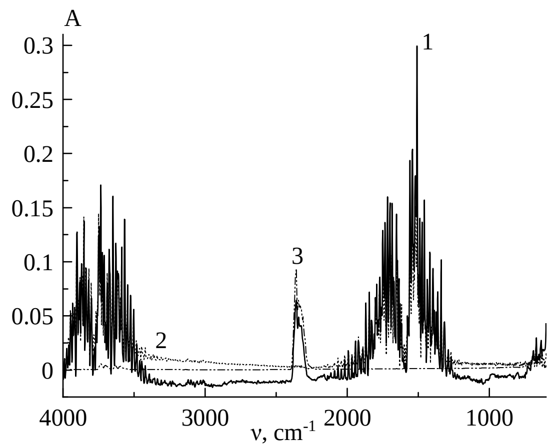


Fig. 4. IR spectrum of gaseous products of ECIG liquid pyrolysis in air atmosphere at 216 °C: 1 – experimental, 2 – water [28], 3 – carbon dioxide [28]
Рис. 4. ИК спектр газообразных продуктов термического разложения жидкости для электронных сигарет на воздухе при 216 °C: 1 – экспериментальная кривая, 2 – вода [28], 3 – диоксид углерода [28]

Perhaps, the organic compounds (if they evolve) are oxidized by air oxygen producing water and CO_2.

Rising the temperature to the values higher than 278 °C, we observed the polyhydric alcohol peaks in the IR spectra of ECIG liquid vapors (Fig. 5).

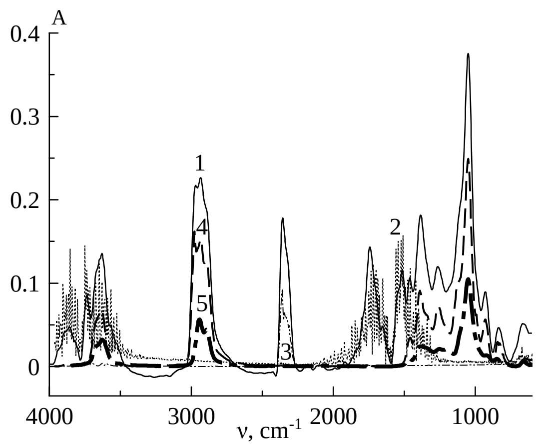


Fig. 5. IR spectrum of gaseous products of ECIG liquid pyrolysis in air atmosphere at 301 °C: 1 – experimental, 2 – water [28], 3 – carbon dioxide [28], 4 – propylene glycol [28], 5 – glycerol [28]
Рис. 5. ИК спектр газообразных продуктов термического разложения жидкости для электронных сигарет на воздухе при 301 °C: 1 – экспериментальная кривая, 2 – вода [28], 3 – диоксид углерода [28], 4 – пропиленгликоль [28], 5 – глицерин [28]

Taking into account the fact that the temperature of electronic cigarette heating unit does not exceed 250 °C [29], the data obtained at lower temperatures only are of some practical interest. In the IR spectra of ECIG liquid at t < 250 °C one could observe water, carbon dioxide, propylene glycol, glycerol and nothing more. Any other impurities were emitted in amounts less than detection limits of method. The probability of acute poisoning of human beings by toxic products of ECIG liquid pyrolysis could be apparently neglected.

It is worth noting that there is a number of articles describing the thermal decomposition of tobacco [e. g. 30-32]. The authors used the experimental conditions and methods similar with ones we have used for studying the electronic cigarette liquid. In the products of tobacco combustion, they have found the large amounts of carbonyl compounds of different structure [30-32], acetaldehyde [31], carbon monoxide [30-32], mixture of phenolic compounds [30, 32], formic acid [30], ammonia [32], hydrogen cyanide [30] and propanal [30]. These toxic components were not detected in the presented study.

CONCLUSIONS

The vapors above heated liquid for electronic cigarettes were studied by IR spectroscopy in the argon and air. Under experimental condition, aqueous vapor, propylene glycol, glycerol and carbon dioxide were detected. No other compounds were found in neither liquid phase by ^{1}H, ^{13}C NMR spectroscopy after heating to 150 °C nor vapors at temperature lower than 250 °C. The probability of acute poisoning of human beings by toxic products of ECIG liquid pyrolysis could be apparently neglected.

In the same time, the danger of the toxic compounds evolving of concentrations lower than experimental method detection limits still exists.

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