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AMMONIUM CHLORIDE INFLUENCE ON REGENERATED CELLULOSE THERMODESTRUCTION PROCESS

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ABSTRACT

Physical and chemical peculiarities of system regenerated cellulose - ammonium chloride thermodestruction process have been studied using thermal and chemical analysis methods. It was defined the dope effect mechanism on the main component destruction depends upon their thermostimulated interaction in the temperature range 180 - 240°C, which leads to nitrogen bonding by solid regenerated cellulose remainder and to separation of the equivalent amount of hydrogen chloride.

INTRODUCTION

Some references in literature can be found /1-5/, devoted to regenerated cellulose (RC) thermal behaviour in ammonium chloride presence. The common deficiency of the above works, to our opinion, lies in that the behaviour of the material is considered only when heating the main component, i.e. RC, while neglecting the doping - ammonium chloride. The gap is partially filled by /4,5/, but still the problem of doping influence on main component thermodestruction is not solved thus being the aim of this paper.

EXPERIMENTAL

In our experiments we used viscose fibre impregnated with aqueous ammonium chloride solutions of various concentration. We studied samples with salt contents 1.0 - 7.0 %, recalculated into nitrogen, prepared and stored as described earlier /4,5/. Experiments were conducted in argon and air ambients under static and dynamic heating modes up to 350°C. TA curves were produced by Q-Derivatograph (Hungary), IR spectra taken by Specord 75 IR (GDR) and X-ray patterns made by DRON-2 machine (Cu Kα-radiation). Nitrogen and chlorine contents in solid residues and gaseous products was defined after their transformation into solution and subsequent chemical analysis.

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RESULTS AND DISCUSSION

From thermal analysis of RC samples with various ammonium chloride contents (Fig.) we can see, that peak shape, number and intensity, as well as their distribution along the temperature range, depend substantially upon the quantity of introduced ammonium chloride as well as upon the ambient where the treating took place. Other experimental factors influence - sample amount, heating regime etc. - is in fact similar to other solids, that decompose by heating with gaseous products emission. It is also seen from Fig. that exact correlation exists between DTA and DTG sample curves, representing interrelation of thermal effects observed with sample mass loss.

The study of nature of processes responsible for the above effects on TA curves with the help of chemical analysis of solid and gaseous pyrolysis products showed that depending on original ammonium chloride contents in RC the former may occur in "active" (up to 14% contents of NH_4Cl in RC) and "inactive" (under higher contents) forms. The decomposition of ammonium chloride "active" form takes place in the 180 - 240°C range, which is significantly lower the pure ammonium chloride starting decomposition temperature and is accompanied by both system components interaction. Quantative nitrogen from salt "active" form bonding by solid RC residue, accompanied by appropriate amount of HCl separation, serves a vivid approval of the above process, which is illustrated by table.

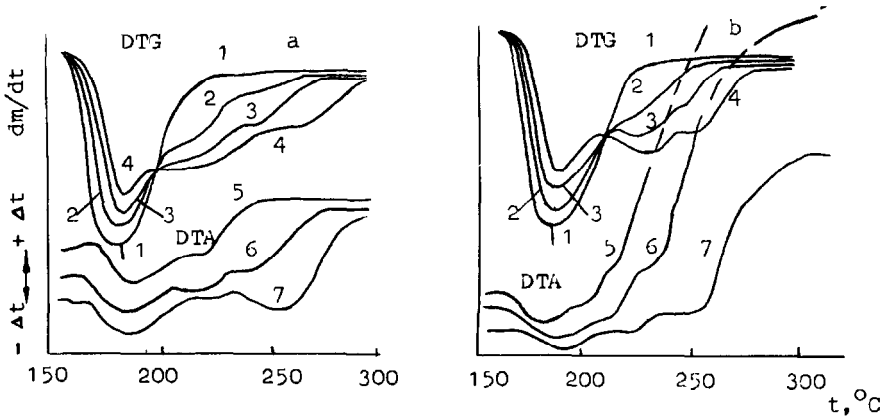

Fig. Curves TA in argon (a) and in air (b) for samples of RC-NH_4Cl system with nitrogen contents,%: 1.0(1); 2.6(2 and 5); 3.7 (3 and 6); 6.8(4 and 7).

"Inactive" part of ammonium chloride in RC starts decomposing at higher temperatures (above 240°C), this process being accompanied by thermal analysis curve change in the vicinity of 250-260°C. "Inactive" form salts quantity in RC samples depends upon the its initial contents and is illustrated by chemical analysis results, given in the table. This salt form presence in thermally processed samples is distinctly established by X-ray analysis and IR spectroscopy.

That is why RC thermally treated in ammonium chloride presence is accompanied by a number of sequential and parallel chemical processes, the main of which in the temperature range discussed (up to 400°C) is the chemical interaction of both components with the formation of reaction products, crystal RC structure destruction and X-ray amorphous carbon residues formation, the latter residues having high carbon contents.

TABLE

Products of RC-NH_4Cl system thermal treatment in argon during 1 h. (In equivalents per RC elemental units in initial sample).

t, °C	NH_4Cl initial	NH_4Cl* "active"	HCl gas	NH_4Cl "inactive"
250	0.340	0.324	0.308	0.035
250	0.535	0.425	0.410	0.135
250	0.754	0.524	0.532	0.230
250	0.989	0.568	0.551	0.422
250	1.176	0.624	0.632	0.550
270	0.672	0.503	0.475	0.195
270	0.892	0.543	0.527	0.365
270	1.184	0.640	0.632	0.551
300	0.616	0.389	0.405	0.219
300	0.884	0.527	0.550	0.365
300	1.167	0.608	0.601	0.568

*This value calculated on the nitrogen analysis data basis (accuracy ±5.0%) and equivalent to its quantity in solid residue RC.

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