KINETICS OF THERMAL DEHYDRATION OF La(III) AND Yb(III) COMPLEXES WITH ORTHO-VANILLIN OXIME

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The analysis of dehydration of the complexes, [La(CsHsNO3)3.2H2O] and [Yb(CsHsNO3)3.3H2O] for the evaluation of kinetic parameters (Z, E & ΔS°) and mechanism of dehydration by non-isothermal methods are reported. The complexes decompose in three well defined steps involving random nucleation mechanism. First two steps involving the dehydration and the third step the loss of the ligand moiety. The intermediates formed during decomposition were found to be unstable for carrying out any significant studies.

The complexes have been already characterized [1]. The present investigation deal with the mechanism of dehydration of complexes and the evaluation of kinetic parameters of the complexes.

Experimental

Paulik-Paulik MOM derivatograph [2] was employed for recording of simultaneous DTA, DTG and TG curves of the complexes. The samples were heated at the rate of 10 deg/min in a cylindrical heat resistant ceramic crucible, α-alumina was used as the reference material.

Non-isothermal methods: Piloyan-Novikova [3], Coats-Redfern [4] and Horowitz-Metzger [5] were employed to analyze the thermogravimetric data and for establishing the mechanism of dehydration. The graph α-T(K) was also analyzed to support the mechanism of dehydration.

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Results and discussion

[La(C₈H₈NO₃)₂H₂O] complex (Fig. 1) starts decomposing at 373 K and this step continues up to 418 K when a rest appears in the curve. The weight remaining at this stage corresponds to [La(C₈H₈NO₃)·H₂O] indicating the loss of one coordinated water molecule. The complex further decomposes at 418 K and this process continues up to 553 K when again a small distinguishable rest is observed. This corresponds to the complete dehydration of the complex. The next decomposition steps continues up to 893 K corresponding to the loss of the ligand moiety. Beyond this temperature there is no weight loss as the stable oxide La₂O₃ is formed.

Similarly, [Yb(C₈H₈NO₃)₃·3H₂O] complex (Fig. 2) starts decomposing from 333 K to 433 K corresponding to the loss of one coordinated water molecule. Further decomposition from 480 K to 595 K corresponds to the loss of the remaining two coordinated water molecules. There is further loss