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# CATALYTIC DECOMPOSITION OF POTASSIUM CHLORATE

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## Abstract

Thermal decomposition of potassium chlorate in the presence of various additives is studied using thermogravimetric analysis and differential thermal analysis. Catalytic effects of metal oxides with comparable surface areas are compared, and the catalytic effects of a number of nonoxide additives are also studied. The nonoxide additives show catalytic activities similar to the corresponding metal oxides. Metal cations and their electron configurations determine the catalytic activity of various compounds. Metal cations with partially filled *d* shells have the highest activity, transition metal cations with completely empty *d* orbitals are moderated active, and metal cations with completely filled *d* shells or noble gas configurations have minimum activity.

**Keywords:** catalytic activity, potassium chlorate, TG-DTA

## Introduction

Thermal decomposition of potassium chlorate and perchlorate have been extensively studied [1–11]. A number of investigators have studied the effect of metal oxides on the thermal decomposition of potassium chlorate and perchlorate, and compared the relative activity of the metal oxides [5–10]. Since the metal oxide additives used by previous authors were from different sources, different activity sequences have been reported and inconsistent or even contradictory catalytic mechanisms have been proposed.

Several papers have concluded that only metal oxides with *p*-type semiconductivity can be active catalysts for potassium chlorate and perchlorate decomposition [2, 5, 11, 12]. But other authors have different conclusions. Said *et al.* reported that in order to be an active catalyst, a compound must be a *p*-type semiconductor and be able to change to *n*-type during the decomposition [6]. Shimolawabe *et al.* considered that high conductivity, not the type of semiconductivity, is responsible for the high catalytic activity [7]. Morishima *et al.* [8] and Iwakura *et al.* [9] suggested that catalytic activity is related to electron hop-

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ping. Metal oxides with low activation energy and small band gap between valence and conduction bands have high activity. Feng *et al.* suggested that the metal oxide containing metal cations with half filled or close to half filled d orbitals are likely to have high activities [10].

It is obvious that there is some contradiction on the catalytic mechanism. The effects of thermal history and surface area of the catalysts were not considered in the previous studies when comparing the relative catalytic activity. No compounds other than metal oxides were studied to test the mechanism. The catalytic mechanisms proposed do not explain the catalytic activity of nonoxide metal compounds.

This work is intended to compare the relative catalytic activity of metal oxides with comparable surface areas for the decomposition of potassium chlorate, and to study the activity of nonoxide metal compounds. The catalytic mechanism will be discussed based on the activity data of the metal oxides and nonoxide compounds.

## Experimental

$KClO_3$, $Li_2SiO_3$, $Na_2Cr_2O_7{\cdot}2H_2O$, $MnCl_2{\cdot}4H_2O$, $FeCl_3{\cdot}6H_2O$, $CoCl_2{\cdot}6H_2O$, $CoF_3$, $NiSO_4{\cdot}7H_2O$, $CuSO_4{\cdot}5H_2O$ and $Na_2SnO_3{\cdot}3H_2O$ as purchased were in the form of coarse crystalline particles, and MgO, $Ca(OH)_2$ and $La(OH)_3$ were fine powders.

In order to make high surface area oxides, precursors with relatively low decomposition temperatures were used. $Cr_2O_3$ was prepared by decomposing chromium(III) acetate hydroxide ($Cr_3(OH)_2(OOCCH_3)_7$) at 425°C. $Fe_2O_3$ was prepared by decomposing $FeC_2O_4{\cdot}2H_2O$ at 350°C. $MnO_2$, $Co_3O_4$ and CuO were prepared by decomposing the corresponding carbonates at 450, 350 and 260°C respectively. NiO and ZnO were prepared by decomposing nickel carbonate hydroxide ($NiCO_3{\cdot}2Ni(OH)_2{\cdot}4H_2O$) and zinc carbonate hydroxide ($ZnCO_3{\cdot}2Zn(OH)_2{\cdot}H_2O$) at 450 and 255°C respectively. $ZrO_2$ was prepared by decomposing zirconyl nitrate ($ZrO(NO_3)_2{\cdot}4H_2O$) at 500°C.

The oxide products prepared were verified by X-ray diffraction analysis using a Rigaku D/max-II Diffractometer. Specific surface areas were measured using the multi point technique with a BET Sorptometer. The sample was heated at 150°C in vacuum to drive off adsorbed gases prior to the measurement. Nitrogen was used as the adsorbate.

Each of the additives was mixed with potassium chlorate in a molar ratio of 4%, corresponding to a ratio of one mole metal cations to 24 mole potassium chlorate. Each of the mixtures was intimately mixed by grinding using a mortar and pestle.

Thermal analysis was carried out using a Netzsch Thermal Analyzer Model STA 409. An approximately 100 mg sample was heated to 700°C at 20 deg·min$^{-1}$ in an oxygen stream of 150 ml/min. The thermogravimetric analysis (TG) and differential thermal analysis (DTA) data were recorded simultaneously.

## Results

The metal oxides prepared were analyzed using X-ray powder diffraction as $ZrO_2$, $Cr_2O_3$, $MnO_2$, $Fe_2O_3$, $Co_3O_4$, NiO, CuO and ZnO respectively. Surface areas of $ZrO_2$, $Cr_2O_3$, $MnO_2$, $Fe_2O_3$, $Co_3O_4$, NiO, CuO, ZnO and MgO were 68, 41, 58, 59, 61, 62, 63, 68 and 67 m$^2$/g respectively. All these oxides had similar high surface areas, which provides a good basis for comparing the relative activities. Most of the metal oxides available from chemical suppliers are usually made at high temperatures and have very low surface areas. High temperature treatment lowers the activity, not only by lowering surface area but also by eliminating defects and active centers. Therefore, metal oxides prepared at high temperatures are usually not good catalysts.

Surface areas of the nonoxide compounds were not measured. These nonoxide compounds tend to decompose at relatively low temperatures and produce highly active products or intermediates. Therefore, the effect of initial surface area is relatively unimportant for these nonoxide metal compounds.

The TG profiles of $KClO_3$ with 4% mole of the oxide additives are presented in Fig. 1. Because the onset decomposition temperature depends on the extent of mixing and grinding, and is difficult to determine unambiguously, the temperature at which 50% $KClO_3$ has decomposed (50% DT) is used in comparing the relative catalytic activities. The experimental error in determining the 50% DT is ±2°C, as previously determined by replicating the decomposition of $NaClO_3$ for three times using the same instrument and experimental conditions.

Figure 1 indicates that $Co_3O_4$ is the most active catalyst for the decomposition of potassium chlorate, and the 50% DT of potassium chlorate catalyzed by $Co_3O_4$ is about 315°C. $MnO_2$, CuO, $Fe_2O_3$, and NiO are also very active even though not as active as cobalt oxide. 50% DTs of $KClO_3$ catalyzed by $MnO_2$, CuO, $Fe_2O_3$ and NiO are approximately 339, 342, 355 and 355°C respectively. MgO and ZnO have very little activity. The 50% DTs of $KClO_3$ containing MgO and ZnO are 579 and 585°C respectively compared to the 50% DT of 606°C for uncatalyzed decomposition of potassium chlorate.

The decomposition of $KClO_3$ catalyzed by $Cr_2O_3$ is a three step process. The first step below 300°C is likely the chemical reaction of $Cr_2O_3$ and $KClO_3$ to produce $K_2Cr_2O_7$, KCl and $ClO_2$ rather than a catalytic decomposition [1]. The second step between 360 and 460°C is the decomposition of $KClO_3$ catalyzed by

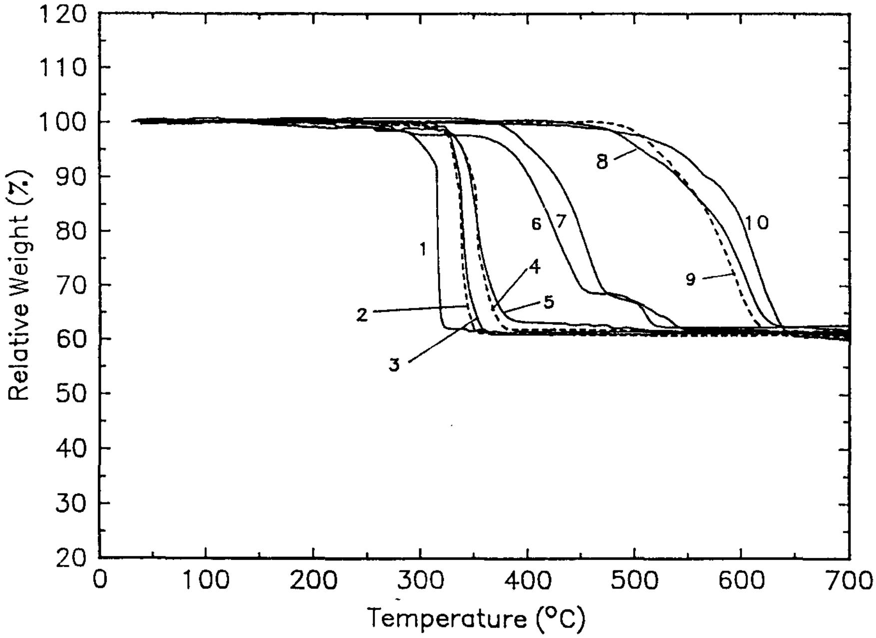

Fig. 1 TG traces of $KClO_3$ catalyzed by metal oxides. (1) $Co_3O_4$, (2) $MnO_2$, (3) CuO, (4) $Fe_2O_3$, (5) NiO, (6) $Cr_2O_3$, (7) $ZrO_2$, (8) ZnO, (9) MgO and (10) no catalyst

$K_2Cr_2O_7$ formed in the first step. The third step between 470 and 550°C is likely the decomposition of $KClO_4$, formed through disproportionation of $KClO_3$, catalyzed by $K_2Cr_2O_7$. It is well known that $KClO_3$ disproportionates during thermal decomposition [10, 13]. $Cr_2O_3$ is green and insoluble in water. The reaction product, however, is brown and water soluble. This is a further indication that the $Cr_2O_3$ has been oxidized to the dichromate. The sample of $KClO_3$ with $ZrO_2$ decomposes at slightly higher temperature than the sample of $KClO_3$ with $Cr_2O_3$.

Figure 2 gives the DTA traces of the samples presented in Fig. 1. The DTA trace of uncatalyzed $KClO_3$ has an endothermic peak at 360°C, and two exothermic peaks at 580 and 630°C respectively. The endothermic peak is assigned to the melting of $KClO_3$ and the peaks at 580 and 630°C due to the decomposition of $KClO_3$ and $KClO_4$. It has been well known that $KClO_4$ can form through disproportionation during $KClO_3$ decomposition [13].

When catalyzed by MgO and ZnO, the melting temperature does not change. Both of the exothermic peaks, however, shift toward the lower temperatures. The peak for $KClO_4$ decomposition is still evident. In the case of $KClO_3$ with $Cr_2O_3$ and $ZrO_2$, the peak for $KClO_3$ decomposition moves further towards a lower temperature and becomes partially overlapped with the endothermic peak. The $KClO_4$ decomposition peak moves to lower temperature and becomes a shoulder of the first exothermic peak.

The intensity of the potassium perchlorate decomposition peak decreases and merges progressively into the background when catalyzed by $Fe_2O_3$, CuO, $MnO_2$, and NiO and disappears completely when catalyzed by $Co_3O_4$. Potassium perchlorate decomposes at much higher temperature even when catalyzed by the most active catalyst cobalt oxide [5]. Therefore, it is unlikely that the peak of $KClO_4$ decomposition has moved further to the lower temperature side and superimposed itself completely with the peak of potassium chlorate decomposition. From top to bottom in Fig. 2, it is clear that an increased proportion of potassium chlorate decomposes directly to KCl and oxygen rather than first disproportioning into $KClO_4$.

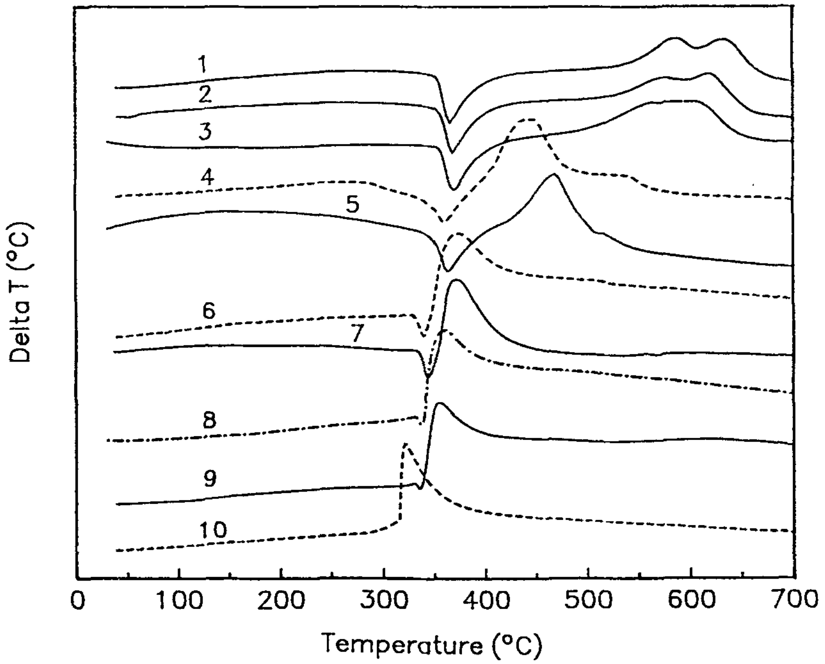

**Fig. 2** DTA of $KClO_3$ catalyzed by metal oxides. (1) No catalyst, (2) ZnO, (3) MgO, (4) $ZrO_2$, (5) $Cr_2O_3$, (6) NiO, (7) $Fe_2O_3$, (8) CuO, (9) $MnO_2$ and (10) $Co_3O_4$

For the samples of potassium chlorate catalyzed by $Fe_2O_3$, CuO, $MnO_2$, NiO and $Co_3O_4$, the exothermic peak caused by the decomposition of $KClO_3$ moves progressively to low temperatures and overlaps increasingly with the endothermic peak, so that the endothermic peak disappears completely for the sample of $KClO_3$ containing $Co_3O_4$. The exothermic peak on the DTA trace of $KClO_3$ with $Co_3O_4$ is located at a temperature lower than the melting temperature of $KClO_3$.

This means most, if not all, of the $KClO_3$ decomposes in the solid state. The melting temperature of $KClO_3$ appears to have shifted to lower temperatures in the presence of NiO, $Fe_2O_3$, CuO or $MnO_2$. This is probably due to the formation of solid solution between the chlorate and the decomposition product chloride [14].

Nonoxide metal compounds can also be very active towards $KClO_3$ decomposition. The TG profiles of $KClO_3$ with 4% mole of $Ca(OH)_2$, $La(OH)_3$, $Na_2Cr_2O_7{\cdot}2H_2O$, $MnCl_2{\cdot}4H_2O$, $FeCl_3{\cdot}6H_2O$, $CoCl_2{\cdot}6H_2O$, $CuCl_2{\cdot}2H_2O$, $NiSO_4{\cdot}7H_2O$, $Li_2SiO_3$ and $Na_2SnO_3{\cdot}3H_2O$ are given in Fig. 3. Even though anhydrous $Na_2Cr_2O_7$, $Na_2SnO_3$ and $Li_2SiO_3$ may be considered as complex oxides, they share the properties of the salts of oxy-acids and are thus compared with nonoxides rather than oxides in this study. Since only electron configurations of the metal cations are of interest in the discussion, it makes no difference to treat them either as oxides or nonoxides. The weight losses below about 250°C shown on some of the curves are due to the evolution of occluded moisture and dehydration of the additives. Figure 3 looks very similar to Fig. 1. This indicates that the nonoxide compounds are parallel in catalytic activity to their corresponding oxides. Oxide and nonoxide compounds of a given metal have

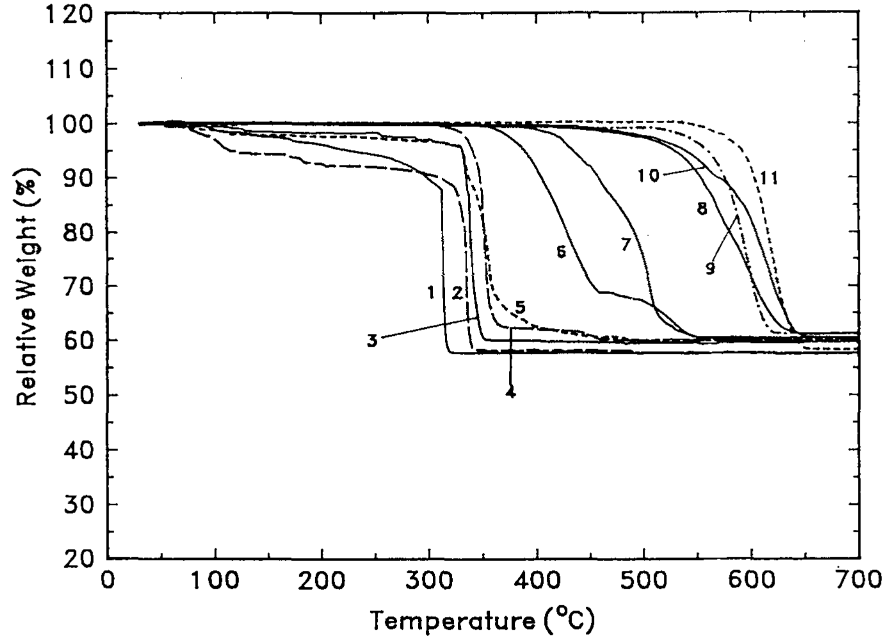

Fig. 3 TG of $KClO_3$ catalyzed by complex oxides and nonoxide catalysts. (1) $CoCl_2{\cdot}6H_2O$, (2) $FeCl_3{\cdot}6H_2O$, (3) $MnCl_2{\cdot}4H_2O$, (4) $CuSO_4{\cdot}5H_2O$, (5) $NiSO_4{\cdot}7H_2O$, (6) $Na_2Cr_2O_7{\cdot}2H_2O$, (7) $La(OH)_3$, (8) $Na_2SnO_3{\cdot}3H_2O$, (9) $Ca(OH)_2$, (10) no catalyst and (11) $Li_2SiO_3$

approximately the same catalytic activity. The TG trace of $KClO_3$ with sodium dichromate looks very much like that of $KClO_3$ with $Cr_2O_3$ as given in Fig. 1. This further indicates the oxidation of $Cr_2O_3$ to the dichromate and that the resulting dichromate may be the actual catalyst for the decomposition of potassium chlorate.

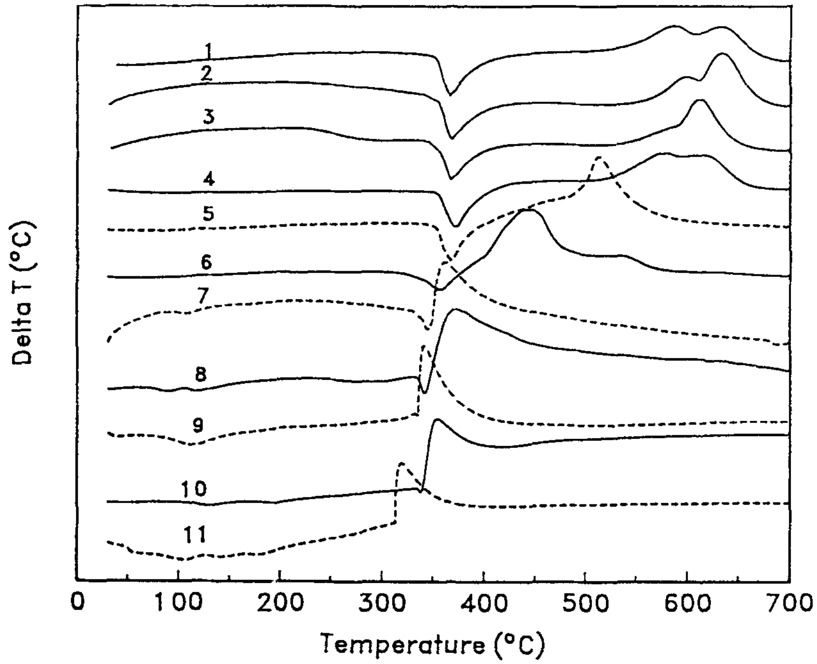

**Fig. 4** DTA of $KClO_3$ catalyzed by complex oxides and nonoxide catalysts. (1) No catalyst, (2) $Li_2SiO_3$, (3) $Na_2SnO_3{\cdot}3H_2O$, (4) $Ca(OH)_2$, (5) $La(OH)_3$, (6) $Na_2Cr_2O_7{\cdot}2H_2O$, (7) $NiSO_4{\cdot}7H_2O$, (8) $CuSO_4{\cdot}5H_2O$, (9) $MnCl_2{\cdot}4H_2O$, (10) $FeCl_3{\cdot}6H_2O$ and (11) $CoCl_2{\cdot}6H_2O$

Figure 4 presents the DTA data for the samples used in Fig. 3. The similarity of Figs 4 and 2 once again indicates that the nonoxide compounds behave similarly to the corresponding oxides.

The sample of potassium chlorate and $Li_2SiO_3$ is a little different from the other samples. In the presence of $Li_2SiO_3$, potassium chlorate decomposes at a slightly higher temperature than the decomposition temperature of potassium chlorate alone. $Li_2SiO_3$ behaves as an inhibitor. The DTA results in Fig. 4 clearly indicate that the second exothermic peak, corresponding to the decomposition of potassium perchlorate, is much higher in the presence of $Li_2SiO_3$. Therefore, $Li_2SiO_3$ cause more $KClO_3$ to disproportionate to $KClO_4$ and thus makes the decomposition temperature higher.

Figure 5 shows that crystal structure and the anion have no significant effect on the catalytic activity. Even though $Co_3O_4$, $CoCl_2$ and $CoF_3$ have different crystal structures and different anions, they have approximately the same activity toward $KClO_3$ decomposition. The close similarity between $MnCl_2 \cdot 4H_2O$ and $MnO_2$ further supports this conclusion.

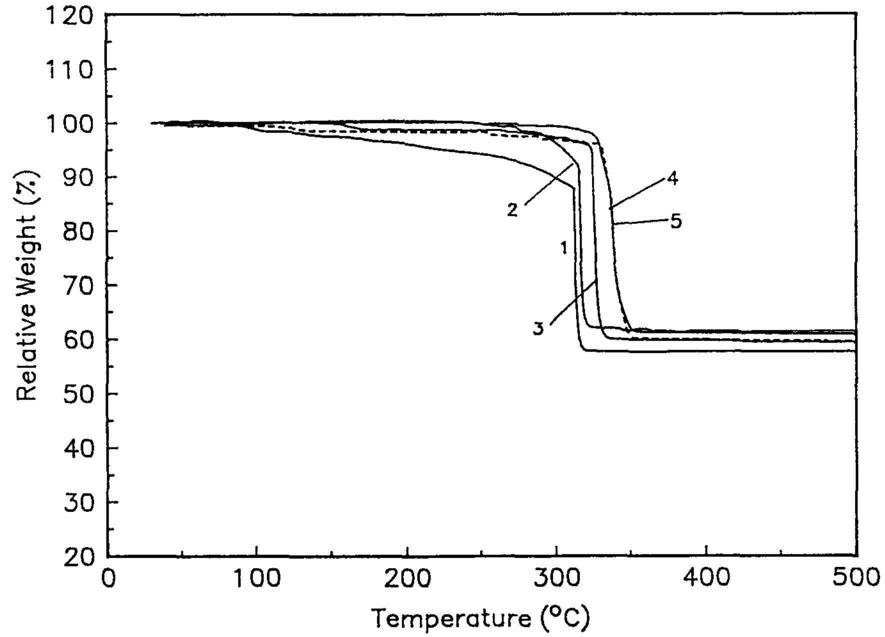

**Fig. 5** TG of $KClO_3$ catalyzed by cobalt compounds and manganese compounds. (1) $CoCl_2 \cdot 6H_2O$, (2) $Co_3O_4$, and (3) $CoF_3$, (4) $MnCl_2 \cdot 4H_2O$ and (5) $MnO_2$

Figure 6 is a comparison of the catalytic activity of three copper compounds. CuCl has considerably lower activity than CuO or $CuSO_4 \cdot 5H_2O$. This is probably related to the difference in their electron configurations, because $Cu^+$ has 10 *d* electrons and has less tendency to form coordination bonds. The higher activity of CuCl compared to ZnO, in which the zinc ion has a 10 electrons in the 3d orbitals, is due to partial oxidation of $Cu^+$ to $Cu^{2+}$ as shown in Fig. 7.

Figure 7 shows the thermal decomposition of $La(OH)_3$, $MnCl_2 \cdot 4H_2O$, $CoCl_2 \cdot 6H_2O$, $CoF_3$, $CuSO_4 \cdot 5H_2O$ and $NiSO_4 \cdot 7H_2O$. CuCl is stable up to about 350°C in oxygen. At about 350°C, it starts being oxidized as indicated by the weight increase. $CuSO_4 \cdot 5H_2O$ loses all five water molecules by about 300°C, and the $CuSO_4$ formed is stable, at least up to 700°C. $NiSO_4 \cdot 7H_2O$ loses the hydration water below 450°C and no further decomposition can be observed up to 700°C. $CoF_3$ is stable up to about 600°C except for a small weight loss below 200°C. This weight loss is probably due to adsorbed moisture. $CoCl_2 \cdot 6H_2O$

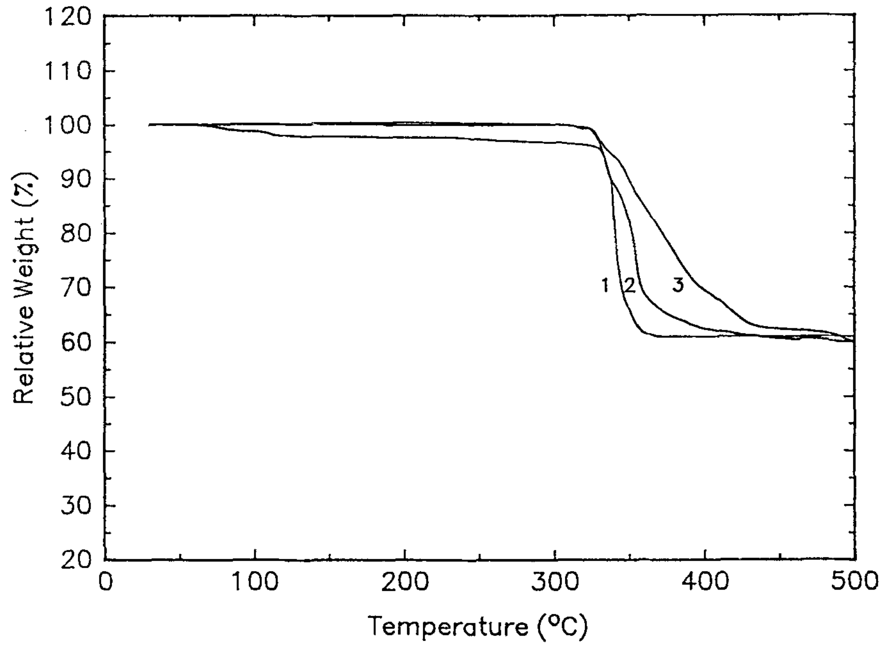

**Fig. 6** TG of $KClO_3$ catalyzed by copper compounds. (1) $CuSO_4 \cdot 5H_2O$, (2) CuO and (3) CuCl

loses all six water molecules below 200°C, and the $CoCl_2$ formed is stable to about 500°C. These results indicate that these nonoxide additives have not been converted to the corresponding oxides before the complete decomposition of potassium chlorate. It may be possible that a very small fraction of the nonoxide compounds has been converted to the oxides even though not detectable by thermal analysis. This small amount of metal oxide, however, would not have significant effect on the decomposition of $KClO_3$, because the catalytic effect increases with increased loading of the oxide catalysts [8].

## Discussion

The *n*-type semiconductor $Fe_2O_3$ has the same high activity as the two *p*-type semiconductors NiO and CuO. Therefore, high activity is not limited to *p*-type semiconductors. Usually changes in crystal structure, oxidation state of metal cations, or the type of anions associated with the metal cations can result in significant difference in semiconductivity because all these factors can cause changes in the band structure. However, as shown in Fig. 5, crystal structure, oxidation state of metal cations, and the type of anions associated with the metal cations have no significant effect on the catalytic activity. Therefore, the catalytic activity is not related to semiconductivity.

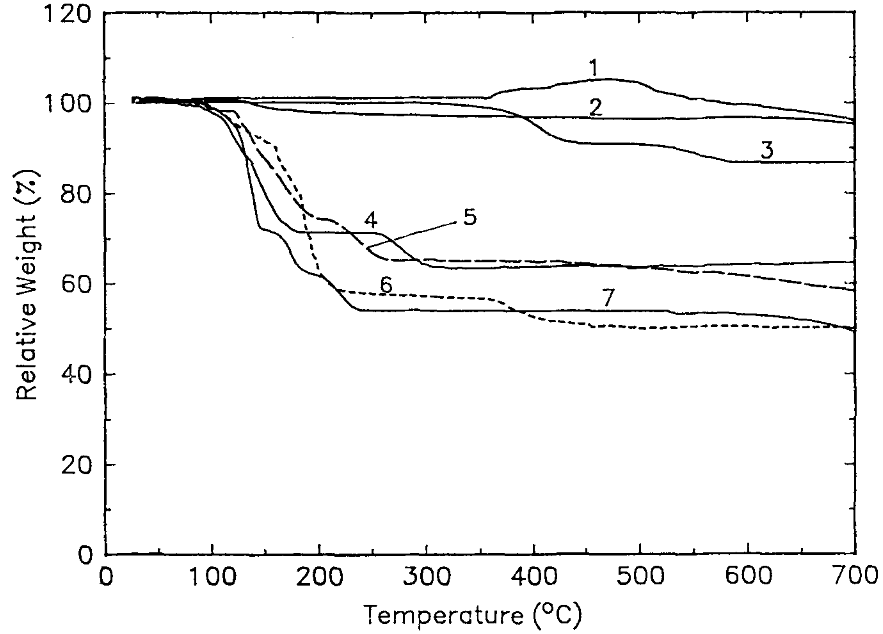

**Fig. 7** TG of nonoxide additives. (1) CuCl, (2) $CoF_3$, (3) $La(OH)_3$, (4) $CuSO_4 \cdot 5H_2O$, (5) $MnCl_2 \cdot 4H_2O$, (6) $NiSO_4 \cdot 7H_2O$ and (7) $CoCl_2 \cdot 6H_2O$

Ionic compounds such as copper(II) sulfate, nickel(II) sulfate and cobalt(III) fluoride are all very active towards $KClO_3$ decomposition. Although there are no electrical conductivity data for these compounds available to our knowledge, it is very unlikely that these compounds are electrical conductors. ZnO has pretty high conductivity but without activity. NiO is an insulator at room temperature but it has very high activity. Therefore, the catalytic activity is not related to electrical conductivity either.

It appears that the identity of the metal cation and its electron configuration determine the catalytic activity of a compound. The electron configurations of metal cations and their catalytic activities are listed in Table 1. It is clear that all metal cations with $nd^m$ ($m$ = 1–9) configurations have high catalytic activity. Transition metal cations with $nd^0$ configurations are moderately active. And metal cations with $nd^{10}$ or noble gas electron configurations have very little activity. This is also consistent with the catalytic effects of some other metal compounds reported. The metal cations of $Gd_2O_3$ and $MoO_3$ both have $d^0$ configuration and they are both moderately active [8]. Compounds containing metal cations with $d^{10}$ configuration, such as $CdCl_2{\cdot}2.5H_2O$ and $Pb(NO_3)_2$, and noble gas configuration, such as $CaCl_2$, LiCl and KCl, all have low activity [3].

**Table 1** Effects of *D*–electrons on activity

| | Cations | Compounds | Activity |
|---|---|---|---|
| $d^2$ | $Cr^{3+}$, $Mn^{4+}$, | $Cr_2O_3$, $MnO_2$ | High |
| $d^5$ | $Mn^{2+}$ | $MnCl_2{\cdot}4H_2O$ | |
| | $Fe^{3+}$ | $FeCl_3{\cdot}6H_2O$, $Fe_2O_3$ | |
| $d^6$ | $Co^{3+}$ | $CoF_3$, $Co_3O_4$ | |
| $d^7$ | $Co^{2+}$ | $CoCl_2{\cdot}6H_2O$ | |
| $d^8$ | $Ni^{2+}$ | NiO, $NiSO_4{\cdot}7H_2O$ | |
| $d^9$ | $Cu^{2+}$ | CuO, $CuSO_4{\cdot}5H_2O$ | |
| $d^0$ | $Cr^{3+}$, $La^{3+}$ | $Na_2Cr_2O_7{\cdot}2H_2O$, $La(OH)_3$, | Moderate |
| | $Zr^{4+}$, $Gd^{3+}$, $Mo^{6+}$ | $ZrO_2$, $Gd_2O_3$, $MoO_3$ | |
| $d^{10}$ | $Cu^+$, $Zn^{2+}$, | CuCl, ZnO, | Low |
| | $Cd^{2+}$, $Pb^{2+}$ | $CdCl_2{\cdot}2.5H_2O$, $Pb(NO_3)_2$ | |
| Noble | $Mg^{2+}$, $Ca^{2+}$, | MgO, $Ca(OH)_2$ | Low |
| gas | $Ca^{2+}$, $Li^+$, $K^+$ | $CaCl_2$, LiCl, KCl (3) | |

The key of the catalytic process is formation of coordination bond between $ClO_3^-$ and the metal cation. A metal cation is a Lewis acid and can accept electron pairs. Each oxygen in the $ClO_3^-$ group has two unshared electron pairs and can donate one pair to the metal cation and act as a Lewis base. Formation of the $O^{2-}$–$M^{n+}$ bond weakens the Cl–O bond in the chlorate and thus results in chlorate decomposition.

Transition metal cations with partially filled *d* orbitals have a higher effective nuclear positive charge because of less electron shielding, and most of these cations have empty *d* orbitals to accommodate extra electron pairs. Therefore, compounds containing metal cations with partially filled *d* orbitals are all active catalysts for the decomposition of $KClO_3$.

The highest activity shown by the cobalt compounds is probably related to the Ligand Field Stabilization Energy. $Co^{3+}$ has a $3d^6$ electron configuration. In an octahedral field, such as in $CoCl_2$, $CoF_3$ and $Co_3O_4$, the five *d* orbitals split into two groups, $t_{2g}$ and $e_g$. Since the lower energy $t_{2g}$ orbitals are completely filled with the six d electrons and the higher energy $e_g$ orbitals are completely empty. $Co^{3+}$ has the highest possible ligand field stabilization energy in an octahedral coordination. Therefore, $Co^{3+}$ has the highest tendency to attract chlorate to form a coordination bond, and the interaction with $ClO_3^-$ would be the strongest. $Co^{2+}$ has a $3d^7$ configuration. But it can be oxidized to $Co^{3+}$.

Cations with $d^0$, $d^{10}$, and noble gas configurations are all spherical. The positive nuclear charge is effectively shielded by the electrons. They have less tendency to attract extra electrons. For metal cations with $d^{10}$, or noble gas configurations, the extra electrons have to go to the much higher outer orbitals. Both of the factors make it unfavorable for these ions to attract extra electrons and to form coordination bonds. Therefore, cations with $d^{10}$ or noble gas configurations are not active. This explains why $Cu^{2+}$ compounds are more active than $Cu^+$ compounds.

For transition metal cations with $d^0$ configuration, on the other hand, the empty d orbitals can accommodate electrons. Therefore, transition metal cations with $d^0$ configuration are moderately active.

## Conclusions

Nonoxide metal compounds have catalytic activities similar to their corresponding oxides. The catalytic activity of a compound is determined by the electron configuration of its metal cation rather than semiconductivity or conductivity. Metal cations with partially filled *d* shell have the highest activity. Transition metal cations with completely empty valence *d* shell are moderately active. And metal cations with completely filled valence *d* shell and noble gas configurations have little activity.

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**Zusammenfassung** — Mittels TG und DTA wurde die thermische Zersetzung von Kaliumchlorat in Gegenwart verschiedener Zusätze untersucht. Der katalytische Effekt von Metalloxiden mit vergleichbaren Oberflächengrößen wurde miteinander verglichen und auch der katalytische Effekt einer Anzahl Nichtoxid-Additive untersucht. Nichtoxid-Additive zeigen ähnliche katalytische Aktivität wie die entsprechenden Metalloxide. Metallkationen und deren Elektronenkonfiguration bestimmen die katalytische Aktivität verschiedener Verbindungen. Metallkationen mit partiell besetzten *d*-Schalen besitzen die höchste Aktivität, Kationen der Übergangsmetalle mit vollkommen leeren *d*-Schalen besitzen eine mittlere Aktivität und Kationen mit vollständig besetzten *d*-Schalen oder mit Edelgaskonfiguration weisen die niedrigste Aktivität auf.