Three Possible Ways to Inhibit the Ammonium Perchlorate Combustion Process

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Proceeding from the assumption that the ammonium perchlorate (AP) combustion process is controlled by dissociation into ammonia and perchloric acid, three possible ways of decelerating the deflagration are considered: a) adding the substances readily generating in decomposition of the ammonia in order to shift the equilibrium of the dissociation to the left; b) adding the substances which can bind the perchloric acid or products of its decomposition into less reactive compounds than perchloric acid itself or original AP; and c) adding the substances which can inhibit the decomposition of perchloric acid. In all cases, the burning rate must decrease in consequence of the reduction in the oxidation rate of ammonia. The readily decomposed salts of ammonia, halogenides of the metals, deoxidants, and hexachlorethane were studied as additives. Experiments were made up in a constant pressure bomb in nitrogenous atmosphere over pressure range from lower pressure limit to 1000 atm. It was shown that the addition of 1% of the salts of ammonia or the halogenides of lead or cadmium to AP, for example, reduces R_m by a factor of 3–4 at low pressures. Deoxidants and inhibitors of the $HClO_4$ decomposition are less effective as burning inhibitors. The synergistic effect in deflagration of AP with inhibitors of different types is also studied in the work. It was shown that intensification of the inhibiting effect of the additives takes place when the double additives of ammonium salts and halogenides of metals are added to AP. The addition of the salts of ammonia and deoxidants to AP does not entail the intensification of the inhibiting effect. An explanation of the regularity obtained and the mechanism of synergistic effect is given in the paper.

I. Introduction

THE possible ways of decelerating the ammonium perchlorate combustion process were shown by us earlier.[1,2] The original idea was that a primary step in AP deflagration is dissociation into NH_3 and $HClO_4$. The readily decomposed salts of ammonia, the excess of which shifts the equilibrium of dissociation to the left, were the most effective inhibitors of the burning.

In the present paper, the effect of the inhibitors (salts of ammonia, halogenides, and deoxidants) on the combustion process was investigated in detail. The effect of the chemical nature of the additive and its quantity on characteristics of the burning; R_m, the coefficient B, and exponent v in the equation $R_m = B_p$, was studied. The experiments were made in a constant pressure bomb, using the method described elsewhere.[3] The particle size of the additives used was $<100\ \mu$, AP $<250\ \mu$, and the quantity of the additives was 1–5% by weight. When the quantity of the additive was less than 5%, the experiments were performed in Plexiglas tubes covered by perchlorvinyl varnish in order to avoid the influence of the material of the tube on the R_m at the pressures above 500 atm.[4]

The coefficient of the inhibition, In, [ratio of mass burning rate R_m (g/cm^2 sec) of pure AP to the R_m of AP with an additive][1,5] is used as a criterion of the effectiveness of the additives. The experiments were set up within a wide pressure interval, from the lower-pressure limit up to 1000 atm. The synergistic effect in the deflagration of AP with inhibitors of different types also was investigated.

II. Experimental Results

As was noted earlier, the effect of the burning inhibitors is not only the decrease of R_m, but also the increase of the lower-pressure limit (LPL), the pressure at which AP starts to burn at given conditions of the experiments. Therefore, the meanings of LPL, In, B, v, and p (the pressure interval over which the burning equation is valid) are given in the tables. Table 1 shows the influence of the salts of ammonia studied in the present work on the burning characteristics of AP. We see that inhibiting effect increases with an increase in the additive content and reduces, as a rule, with pressure. The decrease of effectiveness of the salts of ammonia, with a reduction in the quantity of ammonia generated during decomposition of the salt, is illustrated by Fig. 1. For the salts of hydrohaloid acids, the effectiveness decreases from flourine to bromine.

It is necessary to note the influence of the different metals introduced into the salt on its inhibiting effect. The comparison of the corresponding additives in analogous effect. The comparison of the corresponding additives in analogous conditions shows that the presence of a boron in a molecule of the salt (see additives 1 and 3) for example, strongly reduces the inhibiting effect of the additive. Similar results occur for 5% NH_4F and 5% $(NH_4)_2SiF_6$ (see additives 2 and 4). It is known that some phosphorous salts of ammonia[6] effectively suppressed the first exothermic reaction of AP decomposition and weakly decreased the burning rate of the AP propellants.[7] During the deflagration of the pure AP, as it is seen from Table 1, the ammonium-diphosphate is more effective as an inhibitor of the deflagration. However, ammonium fluoride is the most effective inhibitor and when 1% of it is added to AP it begins to burn at only 100 atm and deflagrates 3.7 times more slowly than pure AP. The halogenides of the different metals and deoxidants which can combine perchloric acid or products of its decomposition also

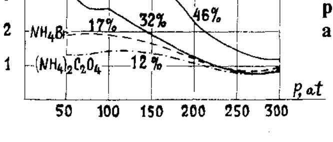

Fig. 1 Inhibition of the ammonium perchlorate (AP) by the effect of ammonia content in the salt (quantity at curves) and pressure.

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Table 1 Influence of readily decomposed salts of ammonia on deflagration of ammonium perchlorate

No. Additive and its contents	LPL, atm	*In*, at the following *p*, atm						*B*	*v*	*p*, atm
		50	100	150	300	600	1000			
1) 1% NH_4F	100	...	3.7	3.6	1.2	0.8	1.1	0.097	0.4	100–200
								0.00011	1.70	200–700
								0.013	0.937	700–1000
2) 5% NH_4F	150	...	...	3.0	1.6	2.8	3.2	0.0033	1.40	200–1000
3) 1% NH_4BF_4	50	1.5	1.1	1.1	1.3	1.0	0.9	0.0181	1.0	50–100
								plateau		100–300
								0.00051		300–1000
4) 5% $(NH_4)_2SiF_6$	200	...	...	...	2.1	1.7	1.2	0.0000012	2.34	200–1000
5) 1% NH_4CI	50	3.3	2.7	1.9	0.8	0.8	0.9	0.0085	0.992	50–500
								0.0019	1.25	500–1000
6) 3% NH_4CI	100	...	3.3	3.1	1.3	0.9	0.9	0.037	0.605	100–200
								0.000054	1.81	300–1000
7) 5% NH_4CI	150	...	...	4.5	1.5	1.8	1.3	0.00015	1.60	200–1000
8) 1% NH_4Br	50	1.9	1.9	1.7	0.8	0.7	0.9	0.09	0.93	50–200
								0.0012	1.33	200–1000
9) 1% $(NH_4)_2C_2O_4$	50	1.3	1.4	1.4	1.0	0.7	0.9	0.225	0.39	50–300
								0.00047	1.45	300–1000
10) 5% $(NH_4)_2C_2O_4$	150	...	...	3.9	1.3	2.1	1.4	0.005	0.99	150–500
								0.000007	2.04	500–1000
11) 1% $NH_4H_2PO_4$	50	2.0	1.8	1.4	0.8	0.9	1.0	0.04	0.717	50–400
								0.00051	1.437	400–1000
12) 1% $(NH_4)_2HPO_4$	50	3.7	2.9	2.3	1.0	0.9	1.1	0.079	0.96	50–400
								0.00071	1.36	400–1000

were investigated as additives. The effect of the additive content on the inhibiting action was studied for AP with lead chloride. According to Adams et al.[8] a very small amount of lead chloride decelerated the deflagration of AP. Figure 2 represents the data obtained.

Table 2 contains the experimental data on the influence of 1% of the salts of different metals on the characteristics of the burning of AP. It is seen from the table that the inhibiting effect of the halogenides of the lead, cadmium, and zinc is close to the salts of ammonia; however, the *LPL* in this case is 50 atm. As seen in Table 2, the fluorides of the corresponding metals are more effective than chlorides. The introduction of boron in the molecule of the additive (compare additives 4 and 5, 13 and 15) strongly decreases the inhibiting action. The same effect was observed for the salts of ammonia.

As it was previously noted, the primary reaction in deflagration of AP is its dissociation into NH_3 and $HCIO_4$.[1] Subsequent reactions between them can lead to the formation of nitric oxides as intermediate products. Levy and Friedman[9] have shown that the quantity of nitric oxides (NO_2, N_2O) decreases with an increase in pressure from atmospheric to 140 atm. The source of the N_2O formation, for example, can be the reaction of NO_2 reduction by radical $NH_2^{\cdot}$:[10,11] $NO_2 + NH_2^{\cdot} \rightarrow N_2O + H_2O$. The progression of such reactions was noted in Ref. 9.

Some of the stabilizers of the thermal stability of double-based propellant, which can combine the nitric oxides even at ambient temperature (e.g., diphenylamine) were effective in inhibiting AP deflagration.[1,5] It is known also that ammonia does not react at ambient temperature with chlorine oxides, but reacts first when methane is added.[12]

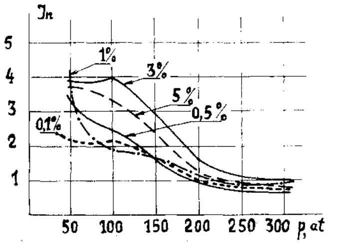

Fig. 2 Inhibition of the AP deflagration by the effect of lead content and pressure.

The amines which contain a small quantity of carbon molecules (such as urea, oxamide, and dicyandiamide) were chosen as deoxidants. According to Sutton et al.,[13] the propellant containing AP as oxidizer burned 1.37 times more slowly when 6% of oxamide was added to it. Table 3 contains the experimental data obtained. As seen from the table, the greater the content of the additive, the higher the LPL. Therefore, for the stoichiometric mixture of AP with urea, the LPL is 600 atm. For *In*, when AP containing 1% of the additive reaches its maximal value at 100–150 atm, the inhibiting action drops. On the other hand at $p > 200$ atm, the value of R_m increases in the presence of these additives. The deoxidants of the aromatic row, many of which were effective inhibitors of RDX deflagration[14,15] also were investigated.

The experimental data obtained show that contrary to data on inhibition of RDX deflagration, amines of the aromatic row are less effective as burning inhibitors than thick-row ones. This is probably because aromatic amines are more easily oxidized by $HCIO_4$ and result in a competitive influence of the additives as the inhibitor and fuel take place. In the pressure region 100–150 atm the inhibiting influence is predominant.

Water and hexachlorethane are effective inhibitors of the thermal decomposition of perchloric acid[15] while C_2CI_6 is also known as a stabilizer of $HCIO_4$. The formation of more stable polyhydrates of $HCIO_4$ is shown in Refs. 16 and 17. According to Levy,[18] the activation energy of $HCIO_4$ decomposition at $t > 315°C$ is 45100 cal/mole, while for aqueous acid it is 84000 cal/mole at 300°C.[19] When 2% of water was added to AP the substance did not burn,[20] even at 150 atm. Table 3 contains data on the influence of 1 and 5% of C_2CI_6 on AP deflagration. At 150 atm, AP with C_2CI_6 burns 1.5 times slower than the pure substance.

Using the analogy of the influence of inhibitors of different types on oxidation of hydrocarbons, it is natural to wait for reinforcement of inhibiting action before adding inhibitors of different classes to AP. The experiments show, however, that by adding 2.5% NH_4F and 2.5% diphenylamine, the effect is not intensified. A similar effect is observed in the AP process with the addition of NH_4CI and C_2CI_6 (see Fig. 3). Another case is observed for AP with salts of ammonia and halogenides. In Fig. 4 and Table 4, it is shown that the addition of an

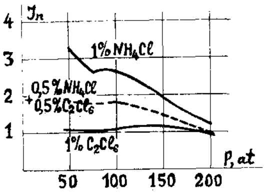

Fig. 3 Curves $In(p)$ for AP with additives NH_4Cl and C_2Cl_6.

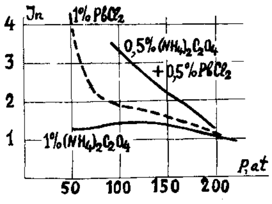

Fig. 4 Curves $In(p)$ for AP with additives $(NH_4)_2C_2O_4$ and $PbCl_2$.

equal weight part of ammonium oxalate, $PbCl_2$, and NH_4Cl with $PbCl_2$ to AP leads to a sharp reinforcement of inhibition. This phenomenon is known as a synergistic effect and reveals itself very clearly on oxidation of hydrocarbons[21] (e.g. ethylbenzene).[22] Because of a radical interaction of inhibitors (amine and phenol types)[23] one of the inhibitors (amine) reduced itself to the original state. In our case the synergistic effect is caused by the simultaneous action on both products of the primary decomposition NH_3 and $HClO_4$. The fact that diphenylamine and C_2Cl_6 were slight inhibitors and there was no reinforce-

Table 2 Meanings of In, B and v for AP with 1% of the additives

	In, at following p, atm								
No. Additive	50	100	150	300	600	1000	B	v	p, atm
							plateau		50–200
1) LiF	0.9	1.4	1.5	1.0	0.9	1.0	0.00051	1.43	300–900
							0.57	0.205	50–300
2) NaF	1.0	1.4	1.4	0.9	0.9	0.9	0.00073	1.33	300–1000
							0.0088	1.325	25–50
3) NaCl	0.7	1.0	1.1	0.9	0.9	1.0	1.15	0.109	50–300
							0.00075	1.38	400–1000
							0.15	0.45	50–300
4) KF	1.3	1.7	1.5	1.0	0.8	0.9	0.002	1.23	300–1000
							0.61	0.21	50–300
5) KBF_4	0.9	1.3	1.4	1.0	0.9	0.9	0.00073	1.38	300–1000
							0.115	0.512	100–300
6) KCl	1.2	1.7	1.6	0.9	0.7	1.0	0.0014	1.29	300–1000
							0.13	0.506	50–200
7) KBr	1.2	1.6	1.4	0.7	0.8	0.8	0.023	0.845	200–600
							0.000052	1.79	600–1000
							0.064	0.647	50–400
8) $K_2C_2O_4$	1.4	1.6	1.4	0.8	0.8	1.0	0.0014	1.29	400–1000
							0.054	0.684	50–500
9) CaF_2	1.4	1.8	1.5	0.7	0.9	1.0	0.0014	1.28	500–1000
							0.53	0.17	50–200
10) $CaCl_2$	1.2	1.8	1.8	1.1	1.0	1.0	0.00032	1.50	300–1000
							plateau		50–100
11) CaO	1.3	2.0	2.0	0.8	0.9	1.0	0.0036	1.148	200–1000
12) ZnF_2	3.7	4.0	3.1	1.0	0.8	1.0	0.0014	1.29	50–1000
13) CdF_2	4.3	4.4	3.1	1.8	0.9	1.0	0.0006	1.40	100–1000
							0.0027	1.17	100–400
14) $CdCl_2$	...	3.3	2.4	0.9	0.7	0.9	0.00037	1.51	400–1000
							plateau		100–200
15) $Cd(BF)_4$	3.3	2.0	2.1	1.1	0.9	1.0	0.0004	1.45	200–1000
16) BaF_2	2.9	2.9	2.2	0.8	0.7	1.0	0.0022	1.22	100–1000
							0.039	0.727	50–400
17) $BaCl_2$	1.9	2.0	1.6	0.7	0.8	0.9	0.00042	1.477	400–1000
							0.31	0.379	50–300
18) HgCl	1.0	1.1	1.1	0.7	0.7	0.9	0.0023	1.225	300–1000
							0.0174	0.867	100–500
19) $AlCl_3$	1.9	2.2	1.7	0.8	0.9	0.9	0.0174	1.52	500–1000
							0.0064	1.11	50–200
20) LaF_3	2.6	2.0	1.3	0.7	0.8	0.9	0.0007	1.41	300–1000
21) ZrF_4	1.9	1.7	1.2	0.5	0.7	0.9	0.018	0.922	50–1000
22) PbF_2	...	2.9	1.8	0.7	0.7	0.9	0.0036	1.16	100–1000
23) $PbCl_2$	4.3	1.9	1.6	0.9	0.8	0.9	0.00034	1.756	50–100
							0.049	0.662	100–300
							0.00074	1.388	300–1000
24) BiF_3	1.6	1.7	1.4	0.7	0.7	0.9	0.042	0.74	50–400
							0.0041	1.74	400–1000
25) CeF_3	1.9	1.4	0.9	0.5	0.6	0.7	0.007	1.14	50–200
							0.0113	1.02	400–1000
26) NdF_3	1.9	2.0	1.7	0.8	0.7	0.9	0.054	0.65	50–200
							0.0032	1.18	200–1000
NH_4ClO_4 without additives							0.068	0.74	50–150
							0.0001	1.67	400–1000

Table 3 Meanings of *In*, *B*, and *v* for AP with deoxidants

No. Additive and its content		LPL, atm	*In*, at following *p*, atm 50	150	300	600	1000	*B*	*v*	*p*, atm
1) Urea	1%	50	2.2	1.8	0.7	0.6	1.0	0.005	1.10	50–1000
	5%	100	...	3.1	1.1	0.8	0.8	0.0003	1.53	100–1000
	30%	...	...	...	...	...	1.2	1.10^{-10}	3.70	650–1000
2) Oxamide	1%	50	1.1	1.6	0.8	0.7	0.9	0.64	0.162	50–200
								0.0008	1.388	200–1000
	5%	250	...	...	1.4	1.0	1.1	0.00012	1.644	250–1000
3) Dicyandiamide	1%	50	1.9	1.5	0.7	0.8	0.9	0.02	0.875	50–600
								0.000092	1.70	600–1000
	5%	50	4.3	2.3	0.8	0.7	0.8	0.0078	0.98	50–200
								0.00086	1.40	200–1000
4) Polyvinylchloride	0.1%	50	1.2	1.3	1.2	1.0	0.9	0.058	0.74	50–100
								5.4	−0.243	100–200
								0.000085	1.70	400–1000
	1%	50	1.3	1.7	1.1	0.7	0.9	0.15	0.482	50–100
								2.2	−0.934	100–200
								0.0002	1.58	300–1000
	5%	50	1.3	1.2	0.9	0.8	0.9	0.05	0.767	50–100
								0.66	0.208	100–300
								0.00105	1.35	300–1000
5) Hexachlorethane	1%	50	1.1	1.2	1.0	1.1	1.0	0.115	0.597	50–100
								plateau		100–400
								0.00004	1.80	500–1000
	5%	50	1.3	1.5	1.2	0.9	0.8	0.200	0.403	50–450
								0.00002	1.93	450–1000

Table 4 Meanings of *In*, *B*, and *v* for AP with double-additives

Additive and its content	*In*, at following *p*, atm 50	150	300	600	1000	*B*	*v*	*p*, atm
2.5% diphenylamine +	...	2.7	1.3	1.7	1.5	0.018	0.78	100–400
2.5% NH_4F						0.0001	1.64	400–1000
0.5% C_2Cl_6 +	1.9	1.5	0.7	0.9	0.9	0.023	0.842	50–700
0.5% NH_4Cl						0.00018	1.59	600–1000
0.5% $PbCl_2$ +	4.3	2.2	1.1	0.9	1.0	0.012	0.858	50–200
0.5% NH_4Cl						0.00044	1.45	200–1000
0.5% $PbCl_2$ +	...	2.2	0.8	0.8	0.9	0.0037	1.124	100–600
0.5% $(NH_4)_2C_2O_4$						0.00018	1.59	600–1000

ment of inhibiting when they were added together, testifies to the assumption that dissociation is rate-controlling in deflagation of AP.

III. Discussion

Let us consider first the dependence of *In*(*p*). As a rule it passes through the maximum at 100–150 atm and then begins to drop, and at $p > 300$ atm, for most of additives investigated, begins to accelerate the AP deflagration. It is connected with the change of rate-controlling reactions with pressure.[2] Once again the data show a conditionality of the concept of catalyzer and inhibitor in deflagration.[2]

The inhibition by the salts of the metals is connected by an exchange reaction they enter with AP.[1,5] The result is the formation of more stable perchlorates of metals[24] than the original AP. The main influence of these additives is that, following decomposition of the salt of ammonia (which is one of the products of the exchange reaction), an additional quantity of ammonia is generated. The excess of ammonia in the reaction zone will shift the equilibrium of dissociation to the left. So, the addition of the inhibitors of different types to AP results in the decrease of the burning rate because of the reduction of the oxidation rate of ammonia.

As for the zone in which the inhibitors affect deflagration, we postulate that a decrease of R_m in the presence of inhibitors is connected with the reduction of the completeness of the chemical reactions taking place in the condensed phase. As seen from the Tables, a sharp decrease of R_m is connected with the diminution of coefficient *B* and the increase of *v* in the burning rate equation. It is necessary to note the influence of the organic inhibitors. The data obtained show once again that the burning rate is not controlled by maximal temperature (T_{max}) of deflagration. If this is so, the addition of organic additives, which act as a fuel to AP, must increase R_m in accordance with an increase of T_{max}. However, this is not so in many cases. Other things being equal, the decisive influence on the burning rate can effect physical properties of the additive, for example its ability to pass into a liquid state,[25] or its chemical properties. In particular, as seen from data obtained in the present work, the ability to bind an active intermediate products of the combustion is important.

Finally, we must pay special attention to the effect of the additives on deflagration and thermal decomposition. Very often there is no such correlation. So, according to Ref. 6, aluminium chloride actually accelerates the decomposition of AP, while in deflagration (see Table 2) it inhibits it. The reduction of $T_{\max}$ by the addition of small quantities of the inhibitors is out of the question. And what is more, in deflagration of AP with paraphormaldehyde $T_{\max}$ increased after the addition of ammonium chloride, according to Arden et al.[26] (The increase of $T_{\max}$ also take place in deflagration of AP with inhibitors of organic nature.)

It might be supposed, however, that the effect of the additives is connected with their influence on thermal processes occurring in the condensed phase. According to the thermocouple estimation of Summerfield,[27] the addition of 1% of LiF to polyurethane propellant reduced the thermal effect in the condensed phase to about 15–20%, and the decrease of burning rate was connected with this fact. However, it is hardly possible that the addition 0.1% of $PbCI_2$, for example, reducing the AP R_m by a factor of 2, can significantly influence the heat release in the condensed phase. Therefore, the deceleration of the AP combustion processes by the additives studied is stipulated first of all by their influence on chemical reactions taking place in deflagration.

Unfortunately, available experimental methods do not permit one to determine the concrete mechanism of chemical reactions in the deflagration of AP with inhibitors. However, as shown in the data, the inhibition of AP combustion processes can be realized most effectively by the influence on the primary act of deflagration—dissociation. And, what is more, it is pertinent to note in conclusion that the idea of AP dissociation in deflagration permits one to eliminate the empirical selection of the burning inhibitors. The examination of this idea showed that deceleration of AP combustion by inhibitors of different types, as well as by use of double additives, can be realized within wide limits.

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