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# INHIBITION EFFECTIVENESS OF DRY CHEMICAL IN METHANE/AIR FLAMES

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**Abstract**—Inhibition effectiveness of dry chemical powders has been examined by detecting the temperature changes in the quenched flat-flame burner. A special particle delivery system was constructed to supply reliable particle concentration to a quenched flat flame. Particle evaporation phenomena in the flat flame environment was investigated by utilizing laser attenuation method which could determine the fraction of particle disappearance in the flame. The result from the flat flame inhibition experiments have clearly demonstrated that the ranking of effectiveness among $KHCO_3$, $NaHCO_3$, and NaCl reverses over the 1200-2000 K temperature range as flame suppressants. The results also showed that dry chemicals are effective inhibitor in the high temperature condition such as in the gaseous flame but not effective one in the coal flame of low temperature. It is concluded that, of all the experiment-specific flame properties, the maximum flame temperature at which the inhibitor evaluation is conducted, was a major parameter to determine the flame inhibition effectiveness in this work.

## INTRODUCTION

Coal-winning and coal and grain handling operations, both underground and on the surface, may be greatly expanded during the next decade to meet World's growing demand for energy and food. Hence, the development of extinguishing techniques for unwanted mine face coal/gas ignitions and grain elevator explosions has become a technical challenge in today's energy and safety conscious civilian world. The rapid efficient suppression of accidental fires in contained environments, such as those in on-board submerged submarines and airborne plane cockpits, also presents an important problem to the military.

Early research for the effectiveness of powdered materials as gaseous flame inhibitors [1-11] established a general ranking of decreasing effectiveness (on a weight basis) among the inorganic agents, as listed in Table 1. Inhibition effectiveness of one inorganic powder is superior to next one by a factor of 2. Dihydrogen ammonium phosphate ($NH_4H_2PO_4$) has an order of magnitude greater in inhibitory effectiveness compared to calcium carbonate. This difference in fire suppression capacity requires the dual classification of materials as being either physical or chemical agents. Physical inhibition is characterized by large quantities of agent required to extinguish a flame, while chemical inhibition was characterized by small amounts of agent needed to retard a flame [12]. All materials usually have both components of this action, with one dominating. The top 7 agents in Table 1 have been routinely classified as chemical agents and the bottom 2 as physical ones.

Quite surprisingly, experimental coal mine dust explosion tests [13-16] revealed that solid chemical agents showed a much altered order of effectiveness compared to what was determined in gaseous flames as in Table 1. For example, $KHCO_3$ was found to be less effective than $CaCO_3$ [15], so that the ranking of effectiveness was almost completely reversed. The experimental results on coal dust explosion suppression independently confirmed such different order of effectiveness in flat flame [17] and jet-stirred reactor [18] studies on pulverized coal combustion.

Explanations [15, 17] for the aforementioned behavior on effectiveness were based on the evaporation rates of the various inorganic chemical powders. If the dry chemical agents listed in Table 1 act as the efficient chemical suppressants, then it is vitally important that the active vapor ingredient be generated early within the flame, thus, it could disturb the normal flame propagation mechanism and cause extinc-

**Table 1. Pre-1973 ranking of relative effectiveness of selected flame suppression agents**

| Agent(s) | Empirical formula | Trade name* | Relative ranking | Reference |
|---|---|---|---|---|
| Potassium bicarbonate + Carbamide | $KHCO_3 + CO(NH_3)$ | Monnex | 1 | 7-10 |
| Potassium bicarbonate | $KHCO_3$ | Purple K | 2 | 2-5, 8-10 |
| Potassium chloride | $KCl$ | Super K | 3 | 3, 6, 8, 9 |
| Sodium bicarbonate | $NaHCO_3$ | BCS | 4 | 1-5, 7, 8, 10 |
| Ammonium chloride | $NH_4Cl$ | – | 5 | 3, 6 |
| Sodium chloride | $NaCl$ | BCD | 6 | 1, 4, 6, 8 |
| Dihydrogen ammonium phosphate | $NH_4H_2PO_4$ | ABC | 7 | 10 |
| Calcium carbonate | $CaCO_3$ | Rock Dust | 8 | 4 |
| Carbon dioxide | $CO_2$ | – | 9 | 2 |
| Water | $H_2O$ | – | 10 | 11 |

*No endorsement is implied. In several of the references cited, the dry chemical agents have been referred to by their trade names. The dual listing given above was done in the interests of clarity.

tion. Reversals of the ranking in inhibitor effectiveness exist within the experimental data of the homogeneous and heterogeneous combustion as well as within the data for each class of flames [17, 19]. Often cited results [20-22] demonstrates that the dry chemicals listed in Table 1 may have no chemical (or homogeneous) action at all. McCamy et al. [2] suggested that their inhibition mechanism was primarily physical since dry chemicals are more efficient than $CO_2$, despite the fact that they only marginally decompose in the flame. From the fact that the direct injection of alkali metals into flames [23] failed to cause any inhibition, conclusion was drawn that the generation of such species is not essential for dry chemicals to possess the high efficiency. This paper offers an alternative interpretation of the McCamy et al. [2]. McHale [24] and Jensen et al. [25] have shown that KOH rather than K is the important gas-phase species in the chemical flame suppression mechanism for potassium-based salts. Iya et al. [20] have suggested that the such assumption is true for the sodium analog.

## STATEMENT OF THE PROBLEM AND APPROACH

The basic research program presented here is designed to circumvent some of the experimental ambiguities from the previous conflicting information on the flame inhibitory capacity of dry chemical agents. A rather innovative approach was carried out for this purpose which is the evaluation of the temperature dependence on dry chemical flame suppression in the simulated fire environments. The versatile quenched flat flame burner [26-28] was utilized to produce an one-dimensional nonadiabatic flame. Whereas the maximum temperature of an unquenched (adiabatic) flame is a unique function of the state of the unburned gas, the maximum temperature of a flame burning on a quenched flat flame burner can be selected and fixed within limits, independent of the properties of the unburned flammable gas. By flowing a combustible fuel/air premixture through the burner at a velocity less than its adiabatic burning velocity, a maximum flame temperature that is lower than its adiabatic flame temperature can be obtained. Proper selection of the fuel/oxidant system and/or the unburned gas velocity provide this nonadiabatic device to generate variable time/temperature histories that would essentially simulate those conditions existing in coal mine gas/air explosions [13-16, 29].

## EXPERIMENTAL

Figure 1 presents a schematic view of the experimental arrangement. Premixed, quenched flat flames were generated on a water-cooled perforated burner which is identical to the one used by Kaskan and coworkers [20, 27]. Six-hundred one millimeter perforations in a rectangular array at the burner surface permitted the introduction of finely-ground dry chemical agents into the flame zone. Quenching of the flame was achieved via cooling coils located beneath the burner outlet and along its perimeter. The maximum flame temperature was varied from 1615 K to 1800 K by changing the cold gas velocity of a stoichiometric methane/air flame. The flame was shrouded with a co-flow of nitrogen in order to prevent entrainment of laboratory air and generation of a secondary diffusion flame. Commercially available methane (99.99 % pure) was used as fuel. Gas flows were controlled by a system consisting of precision gas cylinder regulators in series with a network of nullmatic pressure gauges and critical flow orifices. The critical flow orifices were sapphire watch-maker's jewels set in brass

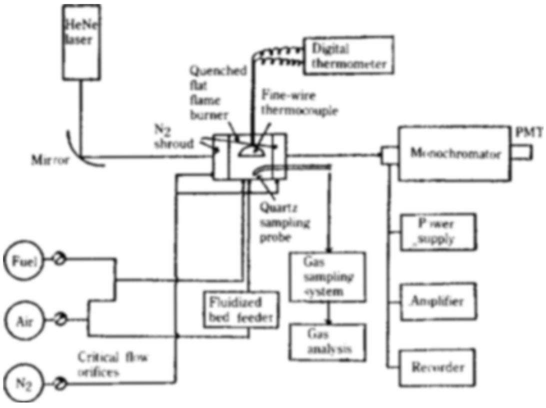

**Fig. 1. Schematic top view of quenched flat flame-Dry chemical inhibition facility.**

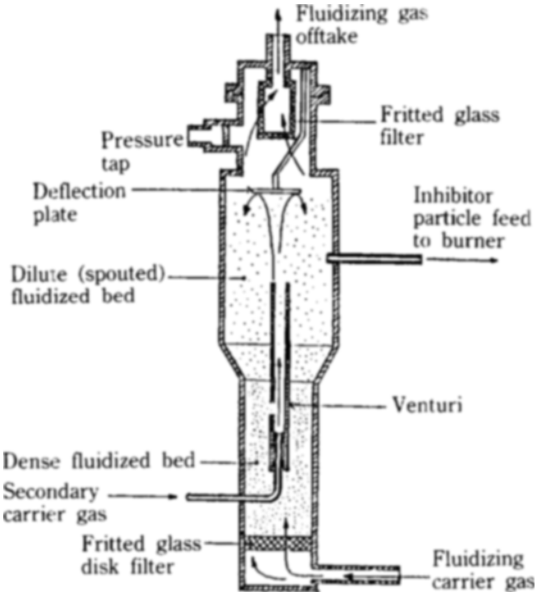

**Fig. 2. Schematic side view of spouted fluidized bed-dry chemical inhibitor delivery system.**

discs. Flow rates were calibrated with a wet test meter.

Flame temperatures were measured by using a silica-coated, $2.7\times10^{-2}$ cm diameter butt-welded Pt/Pt+10% Rh thermocouple. Wire temperatures were calibrated for radiation loss using a revised version [30] of a method first introduced by Kaskan [26]. The dry chemical particle delivery system consisted of a spouted fluidized bed feeder, shown in Figure 2. The particular design offers the following advantages: 1) inexpensiveness of construction, 2) straightforwardness of fabrication, 3) wide range of particle flow rates, 4) simplicity of operation and 5) reproducibility of the results. The particle feeder system was constructed of glasses. Mass flow rates were measured by suspending the entire fluidized bed assembly from a precision balance and monitoring its weight loss per unit time. The pressure drop across the fluidized bed's distributor plate was used to control fluidizing velocity and, therefore, mass flow rate, which ranged 5-100 mg/min. The temperature dependence on inhibitor effectiveness for three different dry chemical agents was studied: $KHCO_3$, $NaHCO_3$ and NaCl. Each inorganic powder was siliconized to improve dispersability. The average surface area of the agent particles was determined as 1550 cm$^2$/g. Particle evaporation phenomena was investigated after introduction of agent into the flame by the use of a visible laser detection system. Red emission (6328 A) from a HeNe laser was attenuated when agent particles still existed in the flame front and ceased to do so after they had been completely evaporated.

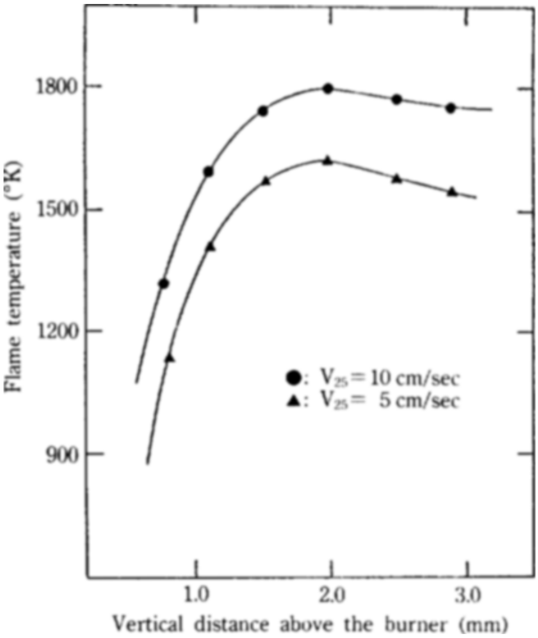

**Fig. 3. Spatial temperature profiles for uninhibited stoichiometric, atmospheric-pressure methane/air flat flames burning different cold velocity.**

## RESULTS AND DISCUSSION

Temperature was the primary independent variable in this work to evaluate the flame inhibition effectiveness of dry chemical agents; residence time was the

**Table 2. Calculated values of particle residence times and heating rates for the stoichiometric methane/air flames studied**

| Cold gas velocity (cm/sec) | Maximum flame temperature (K) | Residence time (m/sec) | Particle heating rate (K/sec) |
|---|---|---|---|
| 5 | 1615 | 12.2 | $1.3 \times 10^5$ |
| 10 | 1800 | 5.1 | $3.5 \times 10^5$ |

secondary one. Changes in temperature upon the addition of agent were used to characterize the extent of inhibition. Figure 3 presents typical spatially-resolved temperature profiles for the coolest and hottest stoichiometric methane/air flames in which inhibitors were evaluated. It shows that as the cold flow velocity was increased, the maximum flame temperature was also increased. The maximum flame temperature remained fixed at a position of 2 mm above the burner surface.

Theoretically-derived temperature profile was first developed with the concept of Mallard and Le Chatelier [31]. They proposed that it was a propagation of heat back through layers of unburned gas that was the controlling mechanism in flame propagation. Unfortunately, this thermal theory requires the concept of a specific ignition temperature, which is still experimentally ill-defined, and neglects the effect of diffusion of molecules, free radicals, and atoms. Theories for particle diffusion mechanisms were first put forth in 1934 by Von Elbe and Lewis [32]. A unit mass passing through the combustion wave at first receives more heat by thermal conduction from the hotter downstream elements than it loses to the upstream cooler elements, so that its temperature increases above the initial level. At higher temperature, the mass element is transformed from a heat sink to a heat source in the sense that it now loses more heat to the upstream elements than it receives from the downstream elements. Its temperature continues to increase, owing to the chemical heat evolution, until the supply of chemical energy is exhausted. In this process, reaction zone thickness is typically characterized as the region from the point where the heat to the upstream elements equals the heat from downstream elements, i.e. ignition occurs, to the point where the chemical reaction is completed. With this definition of reaction zone thickness in mind, the thickness of the flames under investigation was determined in the temperature profiles of Figure 3 as being about 1 mm, independent of cold gas velocity. The same was true for the preheat zone thickness. This spatial resolution of flame events was used without further refinement for calculating particle residence times.

The residence time of a particle in the flame was calculated using the following equation:

$$t_R = \frac{2d}{V_{25}(T_f/T_o + 1)} \tag{1}$$

The combined thickness of the preheat and reaction zone rather than just the thickness of the reaction zone was used in the residence time calculation because the heat required to evaporate particles can diffuse back into the preheat zone from the flame zone under these experimental conditions. Calculated values of the residence times are listed in Table 2 as a function of cold gas velocity and flame temperature. The temperature in Eq. (1) indicates the maximum flame temperature, as measured at the end of the reaction zone. Also included in Table 2 are the calculated particle heating rates, which were determined to be very rapid, and, therefore, could be a minor factor in residence time consideration. Particle heating rates were calculated by the temperature increase of particle within the residence time.

It has been shown that a valid measure of inhibition of a quenched premixed flame burning at a constant unburned gas velocity is a rise in temperature in the inhibited flame, relative to the uninhibited flame [27]. This phenomenon can be understood as follows. When a quenched flame burning at a constant velocity is inhibited, one or more critical reactions are impeded. This caused the flame to burn less closely to the burner, or to lift off slightly, thus reducing the temperature gradient and heat transfer back to the burner surface. As a result, the gas temperatures throughout the flame increase so that presumably the rates of the critical reactions, previously slowed by the inhibitor, increase to the point where they can recontinue to consume the constant throughput of fuel and air. The net reduction in heat transfer then shows up as a temperature rise in the burned gas. The numerical index for evaluating the effectiveness of inhibitors in quenched flames is given by:

$$I^T{}_Q = \frac{T_i^{MAX} - T_o^{MAX}}{[I]} \tag{2}$$

The higher the $I^T{}_Q$, the more effective the flame inhibition agent.

Figure 4 presents the effect of flame temperature on the inhibition effectiveness of $KHCO_3$, $NaHCO_3$ and NaCl. Each dry chemical agent exhibited a different, steep temperature dependency. These data clearly illustrate that the ranking of flame inhibition effectiveness of various inorganic powders is markedly a func-

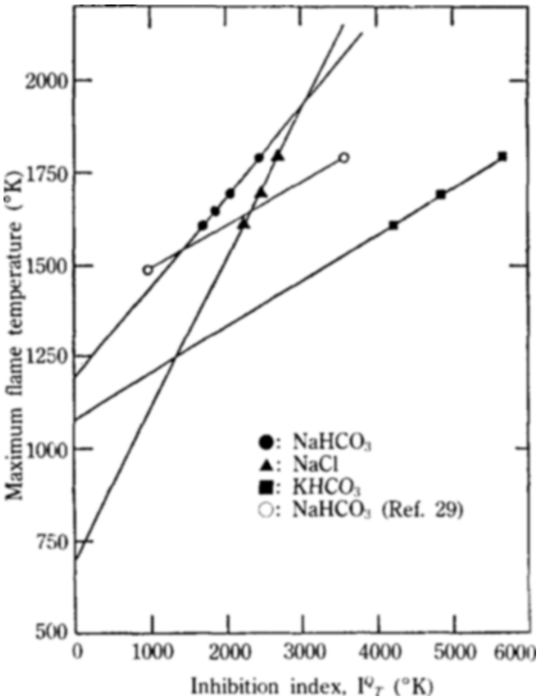

**Fig. 4. Quenched flame inhibitor effectiveness as a function of maximum flame temperature for various dry chemical agents of 20 micron diameter in stoichiometric, methane/air flames.**

tion of the temperature at which the evaluation is conducted. In high temperature region, the ranking according to decreasing inhibitor effectiveness is as follows:

$T_f > 1900$ K: $KHCO_3 > NaHCO_3 > NaCl$ (3)

This ranking matches the aforementioned one (Table 1) determined by other laboratories [2-10] in gaseous flames of temperatures greater than 2000 K. At intermediate flame temperatures, the ranking of decreasing effectiveness appears to be changed:

$T_f = 1250 - 1900$ K: $KHCO_3 > NaCl > NaHCO_3$ (4)

At low flame temperatures, the ranking of decreasing effectiveness apparently becomes:

$T_f < 1250$ K: $NaCl > KHCO_3 > NaHCO_3$ (5)

Most noteworthy was the finding that as the flame temperature was varied from 1200 to 2000 K, the inhibitor capacity of NaCl changed from the best to the worst. Residence time must play a secondary role in determining the relations [1, 2], because it increased two-fold over the temperature range-reduction studied while the effectiveness of each agent diminished.

It should be noted that there is no scientific justification for drawing staight lines through the data point given in Figure 4, although the linear correlation coefficients were quite acceptable. The crossover points and intercepts are illuminating. The data in Figure 4 indicate that NaCl and $KHCO_3$ exchange the superiority in effectiveness at temperature of 1250 K; NaCl is more effective at the lower flame temperatures within the range of this experiment. Results of Richmond et al. [14] revealed that NaCl is superior to $KHCO_3$ at a coal dust/air explosion temperature of 1300 K. The data in Figure 4 also indicate that NaCl and $NaHCO_3$ reverse superiority in effectiveness at 1900 K; NaCl remains more effective at the lower temperatures. Dolan [1] has determined that $NaHCO_3$ is superior to NaCl at temperature of 1700 K. In general, therefore, the crossover data in Figure 4 correspond to data available in the literature [1, 14]. The linear extrapolations in Figure 4 predict that $NaHCO_3$ and $KHCO_3$ become completely ineffective as chemical flame inhibitors at temperatures less than 1200 K and 1100 K, respectively. As such, the data in Figure 4 validate those determined in the experimental mine suppression tests [13-16] which revealed that $KHCO_3$ and $NaHCO_3$ are as ineffective as $H_2O$ at temperature of 1400 K. Moreover, among the three common dry chemical agents studied here and in the experimental coal mine tests [13-16] and jet-stirred reactor studies [18], NaCl was always the most effective suppressant at low flame temperature. Finally, Mitani and Niioka [21] stated that the species responsible for chemical inhibition upon $NaHCO_3$ addition, if it exists, must appear around a temperature of 1127 K, the melting point of $Na_2CO_3$ which is the major decomposition product of $NaHCO_3$. The temperature-dependent effectiveness data for $NaHCO_3$ in Figure 4 extrapolate to 1200 K, not far from this limit temperature for the onset of chemical inhibition [21].

The HeNe laser attenuation measurements for the entire temperature range (1615-1800 K) revealed that the 20 micron particles of each agent had completely evaporated before the plane of the maximum temperature in the premixed flat flames. Iya et al. [20] also observed this effect at 1800 K for methane/air flames inhibited by similarly-sized $NaHCO_3$ powder. At cooler temperatures, the inhibitor capacity diminished because of incomplete evaporation of dry powders. McCamy et al. [2] determined that only 15% of powdered $NaHCO_3$ agent had decomposed in their flame suppression tests; although they did determine that $NaHCO_3$ was a more effective suppressant than $CO_2$ with the margin of factor 2. The data in Table 1 were determined at flame temperatures of greater than 2000 K with a margin of 100s. This apparent discrepancy can be explained by the rather cool flame temperature experiments of McCamy et al. [2]. Mitani and Niioka [21] studied the inhibition of $H_2/O_2/N_2$ flames by variously-sized $NaHCO_3$ powders, both experimentally and theoretically. They used the classic burning-velocity reduction measurement at a constant temperature for the evaluation of $NaHCO_3$ inhibitor effectiveness in adiabatic premixed flames. With their data, one can evaluate $NaHCO_3$ inhibition effectiveness as a function of temperature, as was done in this study using the temperature-rise measurement of inhibition for constant-velocity nonadiabatic premixed flames [20]. These Mitani and Niioka [21] data are also plotted in Figure 4 as open circles. The temperature dependence for $NaHCO_3$ on flame inhibition directly determined in this study and that extracted from the data of Mitani and Niioka [21] are not much different given the fact that experimental conditions of two studies were quite different, i.e., different fuels ($CH_4$ vs. $H_2$), different flames (quenched vs. adiabatic), and different size of $NaHCO_3$ powders (20 vs. 54-17 microns).

In closing, a few words are necessary regarding the absolute values of $I^{T}_{Q}$ presented in Figure 4. Because HeNe laser attenuation experiments revealed that the 20 micron particles of all three agents had completely evaporated within the flame zone, it could be deduced that the active agent for suppression, the alkali metal, had been released in all the cases. It was thus necessary to calculate the measure of effectiveness based on the mole fraction of sodium or potassium delivered to the flame. It is recognized that practical use of inhibition data generally requires effectiveness data expressed on a weight basis.

## CONCLUSIONS

Quenched premixed flat flame inhibition experiments have clearly demonstrated that the ranking of effectiveness among $KHCO_3$, $NaHCO_3$, and NaCl as combustion suppressants reverses over 1200-2000 K temperature range. As flame temperature increases over this range, the inhibitor capacity of NaCl changes from the best to the worst. Therefore, it can be stated that the flame temperature at which an inhibitor evaluation is conducted plays an important role in determining the fire-fighting capacity of the specific dry chemical agent. The different and steep temperature dependencies for inhibition determined for the three agents can explain why previous flame inhibition experiments have not produced apparently conflicting and confusing information on the relative ranking of effectiveness of a variety of flame suppression agents. Since data in Table 1 have been assembled irrespective of their experiment-specific flame properties (premixed /diffusion; laminar/turbulent; one-/three-dimensional; adiabatic/nonadiabatic; gaseous/solid fuels), the maximum flame temperature clearly appears to be the common denominator.

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## NOMENCLATURE

$t_R$ : residence time [sec]
d : thickness of the preheat zone plus reaction zone [mm]
$V_{25}$ : cold gas velocity [cm/sec]
$T_o$ : ambient temperature [K]
$T_f$ : flame temperature [K]
$I^{T}_{Q}$ : inhibition index for quenched flame
$T_i^{MAX}$ : inhibited flame maximum temperature [K]
$T_o^{MAX}$ : uninhibited flame maximum temperature [K]
[I] : inhibitor mole fraction

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