Burning-Rate Enhancement of Organic Diazide Propellants: Dihalide Addition and Pressure Elevation

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Following the recent development of a class of organic diazides as high-energy-density, high-burning-rate fuels, an experimental investigation on the combustion, vaporization, and microexplosion of droplets of these organic diazides and their mixtures with alkyl halides at atmospheric and elevated pressures has been conducted in an effort to further enhance their high-burning-rate characteristics. Results show that the addition of small quantities of alkyl dihalides leads to significant advancements in the state of droplet microexplosion, regardless of the halogen substituent and its relative volatility, that increasing pressure dramatically increases the droplet gasification rate, and that these enhancements can lead to order-of-magnitude increases in the overall gasification rate of the droplet over those of hydrocarbons. The importance of enhanced liquid-phase reaction due to dihalide addition and droplet temperature elevation is emphasized.

Introduction

ATTEMPTS to boost the power output of high-performance, volume-limited, liquid-fueled rockets and ramjets have traditionally focused on improving the atomization, droplet gasification, macroscale mixing, and gas-phase reaction charcteristics of the overall spray generation and combustion processes. These processes, however, have intrinsic limitations on the extent to which improvements can be achieved. For example, ultrafine droplets cannot be easily generated and are probably also not desirable from macromixing considerations. As far as droplet gasification is concerned, increases in the gasification rates are limited by the intrinsic heat and mass transfer rates to and from the droplet. Within this framework, then, potential increases in the droplet gasification rates are at most a few tens of percent.

Recently, a class of organic diazides has been developed[1,2] that holds the potential for improving the performance of jet fuels. These diazides exhibit remarkably fast droplet gasification rates as well as strong propensity to microexplode—an event that has been termed secondary atomization. They have the generic structure of N_3-R-N_3, where N_3 is the azido group and R is a linear alkyl group, $-(CH_2)_n-$, with n being the carbon chain length. While the energetics of the molecule is primarily determined by oxidation of the alkyl group, thermal decomposition of each azido group to a nitrene radical and molecular nitrogen[3,4] yields roughly an additional 50 kcal/mole of heat. The decomposition can occur in either the liquid or gas phase depending on the prevailing temperature.

Figure 1 compares the d^2-law droplet burning- and vaporization-rate constants K for alkyl diazides and unsubstituted alkanes. It is seen that whereas the burning-rate constants of alkanes are around 1 mm^2/s, the burning rates of diazides can exceed these values by up to a factor of 7 for the diazides investigated. This significant increase is most pronounced for the lighter molecules and is not limited to burning because the

same extent of enhancement is also observed for pure vaporization in hot, inert environments. Furthermore, pure diazide droplets also exhibit strong microexplosion events characterized by intense and early droplet disruption, which are expected to rapidly lead to complete liquid gasification. The efficiency of microexplosion can be quantified by the droplet diameter at microexplosion d_e normalized by its diameter at ignition d_o, as shown in Fig. 1 for the burning situations. It is seen that the droplet lifetime can be significantly shortened by the occurrence of microexplosion, especially for the heavier molecules.

It is clear that this significant extent of gasification rate augmentation, as well as microexplosion by an initially pure material, cannot be explained by conventional droplet gasification and microexplosion theories.[5-7] The unique feature here is the occurrence of liquid-phase reactions within the droplet as it is heated up during gasification. The subsequent chemical heat release, though small in quantity as compared to that from gas-phase oxidation (e.g., ~900 kcal/mole for diazidopentane[8]), is significant compared with the latent heat of vaporization (e.g., ~14 kcal/mole for diazidopentane[8]) and, thereby, greatly facilitates the gasification of the liquid fuel. The liquid-phase heat release can also locally raise the liquid temperature to exceed its limit of superheat, leading to instantaneous internal gasification and, consequently, droplet disruption. It is also significant to note that these beneficial effects were not observed for monoazides, whose behaviors are similar to those of the conventional hydrocarbons. It appears that, because of the difunctionality of diazido fuels, reactions between diradicals can propagate quickly.[4]

Recognizing that, although the lighter diazides have faster gasification rates, they tend to microexplode later in the droplet lifetime and thus reduce the effectiveness of microexplosion as a means of secondary dispersal of the liquid fuel and that, although the heavier diazides tend to microexplode earlier, they have slower gasification rates. It is then clear that an optimal, high-thrust fuel would possess the fast burning rates of the light diazides combined with an early microexplosion event characteristic of the heavy diazides so as to achieve further reductions in the droplet lifetime greater than what have been observed so far. Thus, the objective of the present work is to develop ways of further enhancing both the rapid burning and microexplosiveness of the diazides. Two approaches have been explored and are demonstrated to be significantly beneficial. In the first approach, chemical additives have been examined that can further facilitate the diazide reactivity with
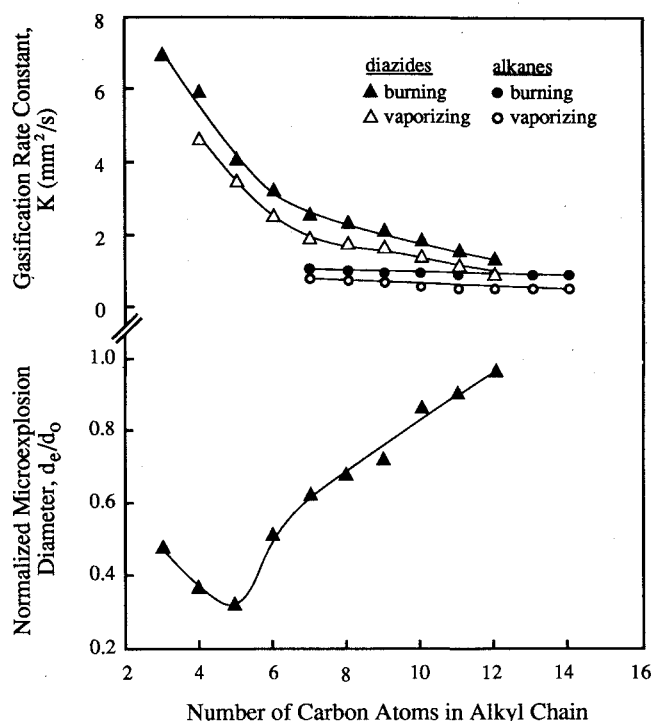
Fig. 1 Comparisons of burning- and vaporization-rate constants, and normalized droplet microexplosion diameters for diazides vs the number of carbon atoms in the alkyl chain, at 1 atm, from Ref. 2.

only a small amount of addition. The successful additives identified are the alkyl dihalides, which have been found to significantly advance the onset of droplet microexplosion. In the second approach, the liquid-phase reaction rate is increased by raising the droplet temperature through elevation of the system pressure. It will be shown that the resulting enhancement in the overall droplet gasification rate due to these individual as well as their combined effects can be substantial.

The experimental methodology will be described briefly in the next section, which is followed by the presentation of the experimental results on dihalide addition, pressure elevation, and the combined effects of dihalide addition with pressure elevation.

Experimental Methodology

The experiment involves downwardly injecting a single stream of monodisperse droplets in a coflowing, hot, gas stream produced from a flat flame burner. An atmospheric pressure combustor and an elevated pressure combustor are used in the investigation. The combustor environment can be varied between nonoxidizing and 21-mole-% oxygen and can be pressurized up to 4 atm for the elevated pressure combustor. The temperature of the combustion gas is about 1050 K for the atmospheric pressure combustor and 1200 K for the elevated pressure combustor, which has better insulation. The droplets, generated by a piezoceramic crystal, are varied in initial size from 200 to 250 μm. Droplet photomicrographs, for droplet size determination, are obtained with a stereomicroscope and a variable time-delay strobe flash synchronized with the droplet generation circuitry. Gasification rates are extracted from d^2-law plots of the experimental droplet size histories prior to the onset of microexplosion. The effectiveness of microexplosion is quantified by the normalized droplet diameter at microexplosion. Further details regarding the experimental assembly and approach can be found in Refs. 2, 9, and 10.

The basic methodology then involves the determination of the droplet gasification rate and microexplosion size under various ambient conditions, with parametric variations of the mo-

lecular structure and quantitative addition of various alkyl dihalides to diazides. Since the entire homologous series of terminally substituted alkyl diazides has already been studied,[2] the present investigation will concentrate on a single representative diazide, 1,5-diazidopentane, so that the effects of dihalide addition and system pressure elevation can be studied more extensively. Diazidopentane has the structure N_3-$(CH_2)_5$-N_3 and is selected because its volatility is similar to those of jet fuels.[8] Furthermore, since it has the smallest droplet microexplosion diameter (Fig. 1) and, hence, the poorest secondary atomization characteristics, it is the most suitable candidate for the study of facilitated microexplosion and enhanced droplet gasification rate. The dihalides in this study have the same terminally substituted form, X-R-X, where R is a linear alkyl group and X = Cl, Br, and I. All referrals to mixture concentrations are in terms of volume fraction or percent.

As a reference, note that the burning-rate constant of dodecane, whose boiling point is close to that of diazidopentane, is 0.89 mm^2/s for the same combustion environment in which the data of Figs. 1–10 are taken.

Dihalide Addition Under Atmospheric Pressure

Although pure organic diazides already exhibit rapid gasification and inherent disruptive behavior, addition of a few percent of terminally substituted alkyl dihalides advances and intensifies the droplet disruption even further. Since thermochemical estimates[8] show that blending of alkyl halides does not provide a sufficient boost in the overall energetics of the mixture to cause catastrophic droplet disruption, a chemically induced mechanism may be likely. Further recognizing that microexplosion of a multicomponent droplet is strongly influenced by the volatility differentials between the constituents,[9,10] two series of experiments have been conducted in which the chemical effects of particular halogen atoms are first independently identified, with the effects of volatility differential subsequently investigated.

Chemical Effects—Equal Volatility Mixtures

First, the chemical effects of the halogen atom substituent are identified by performing experiments with dihalides chosen so that there is, essentially, no volatility differential between the dihalide and the test diazide. This is to eliminate microexplosion or burning-rate variations due to mixture fractionation induced by volatility differential effects.[9,10] Thus, dichlorides, dibromides, and diiodides have been investigated that have similar volatilities with diazidopentane. The dihalides chosen are 1,7-dichloroheptane, 1,5-dibromopentane, and 1,3-diiodopropane. Figure 2 shows the d^2-law burning-rate constant K for these equal volatility diazide/dihalide mixtures as a function of dihalide addition. The results show that, with a small amount of dihalide, the gasification rate K is either unchanged
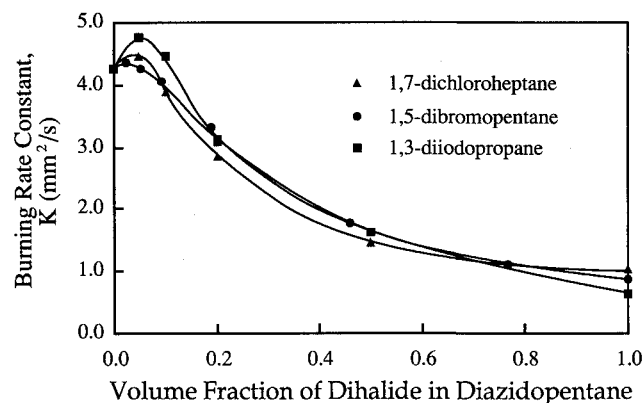

Fig. 2 Burning-rate constant K for mixtures of 1,5-diazidopentane with various amounts of equal volatility dihalides: 1,7-dichloroheptane, 1,5-dibromopentane, and 1,3-diiodopropane, at 1 atm.

or mildly increased. Both the trend and the degree of this marginal enhancement is essentially the same for all of the halides examined, with the iodated compound possibly exerting a slightly stronger response. However, successively higher levels of halide addition rapidly dilute any gain in gasification rate enhancement.

Although the droplet burning rate is only mildly affected by low levels of dihalide addition, Fig. 3 shows that the droplet microexplosion size is significantly increased, by as much as a factor of 2–3 over that of the pure diazide, and can approach a normalized microexplosion diameter of 0.85. Since the droplet mass is proportional to the droplet diameter cubed, nearly 60% of the initial droplet mass is dispersed through microexplosion for 10% addition of dihalide, whereas only 3% of the initial mass is dispersed for the pure diazide without dihalide addition. Thus, microexplosion with dihalide addition is nearly 20 times more effective in the rapid dispersal of the total droplet mass. Furthermore, since the diazide fuels are extremely fast-burning materials, a relatively large diameter at microexplosion also signifies rapid onset of droplet disruption in terms of the actual physical time. As in the burning-rate data of Fig. 2, the trend and level of microexplosion augmentation is essentially the same for all of the dihalides, although again, the iodated compound appears to show a slightly stronger response. As dihalide addition increases, however, the beneficial effects are gradually diluted and result in reductions of both the gasification rate and the microexplosion size. The acceleration of the droplet disruption event by dihalide addition is clear evidence of facilitated liquid-phase reactions in the droplet.

It may be noted that a greatly advanced microexplosion event, as shown in Fig. 3, is more significant in terms of reduction in the droplet lifetime than a moderately increased burning rate, as shown in Fig. 2. This is because the burning-rate constant K is determined only for the pre-explosion period such that its influence is also confined to that time period. For a greatly advanced microexplosion event, this period is rather limited. Since the occurrence of microexplosion is expected to rapidly lead to complete liquid gasification, the reduction in the overall time for gasification is then mostly due to the advanced microexplosion. Thus, dihalide addition results in a significant enhancement of the overall burning rate of the diazide.

To demonstrate this point, we define an overall burning-rate constant $\bar{K}$ as

$$\bar{K} = d_o^2/\tau_e \qquad (1)$$

where τ_e is the droplet burning time up to the state of microexplosion. Since

$$d_e^2 = d_o^2 - K\tau_e \qquad (2)$$

we have

$$\bar{K} = \frac{K}{1 - (d_e/d_o)^2} \qquad (3)$$

Figure 4 plots $\bar{K}$ for the data of Figs. 2 and 3. Recognizing $\bar{K} \approx K = 0.89$ mm^2/s for dodecane, the resultant increase in the burning-rate constant is quite evident.

Mixtures with Volatility Differentials

It is also of interest to investigate whether the droplet gasification response can be further manipulated by using dihalide additives whose volatilities are substantially different from that of the host diazide droplet. Figures 5–8 show the burning-rate constants, the normalized microexplosion diameters, and the overall burning-rate constants for dihalides which have higher and lower volatilities than diazidopentane. The higher volatility dihalides chosen are 1,4-dichlorobutane and 1,3-dibromopropane, which have boiling points approximately 50 K lower

Fig. 4 Overall burning-rate constant $\bar{K}$ for mixtures of 1,5-diazidopentane with various amounts of equal volatility dihalides: 1,7-dichloroheptane, 1,5-dibromopentane, and 1,3-diiodopropane, at 1 atm.
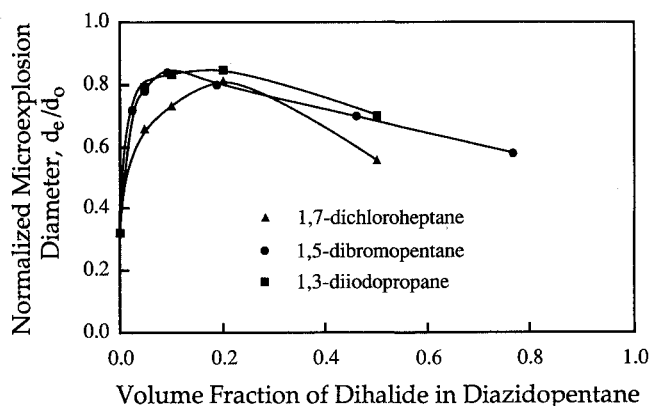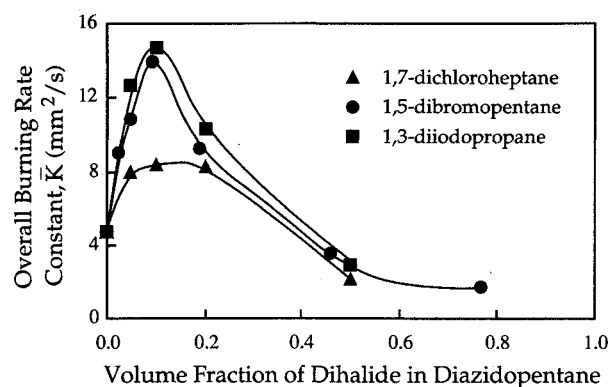
Fig. 3 Normalized microexplosion diameter for mixtures of 1,5-diazidopentane with various amounts of equal volatility dihalides: 1,7-dichloroheptane, 1,5-dibromopentane, and 1,3-diiodopropane, at 1 atm.
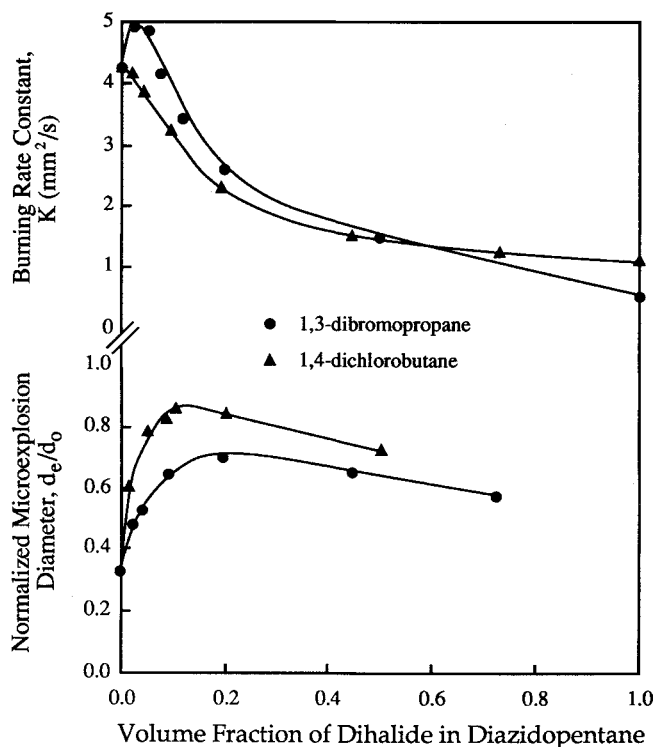
Fig. 5 Burning-rate constant K and normalized microexplosion diameter for mixtures of 1,5-diazidopentane with various amounts of higher volatility dihalides: 1,4-dichlorobutane and 1,3-dibromopropane, at 1 atm.
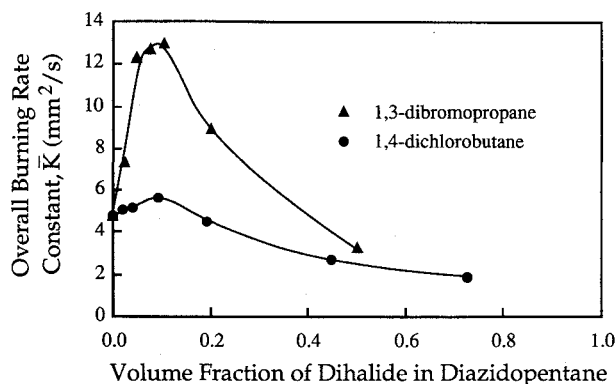
Fig. 6 Overall burning-rate constant $\bar{K}$ for mixtures of 1,5-diazidopentane with various amounts of higher volatility dihalides: 1,4-dichlorobutane and 1,3-dibromopropane, at 1 atm.
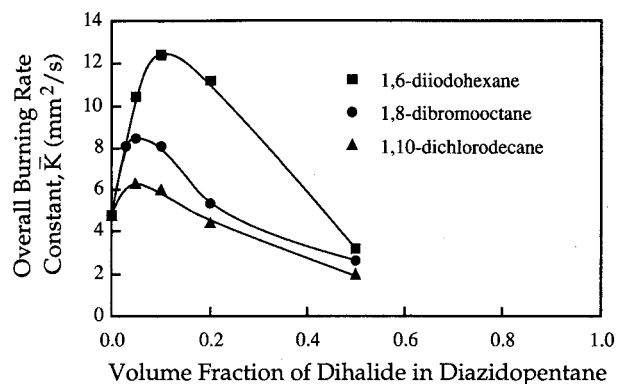

Fig. 8 Overall burning-rate constant $\bar{K}$ for mixtures of 1,5-diazidopentane with various amounts of lower volatility dihalides: 1,10-dichlorodecane, 1,8-dibromooctane, and 1,6-diiodohexane, at 1 atm.

Fig. 7 Burning-rate constant K and normalized microexplosion diameter for mixtures of 1,5-diazidopentane with various amounts of lower volatility dihalides: 1,10-dichlorodecane, 1,8-dibromooctane, and 1,6-diiodohexane, at 1 atm.
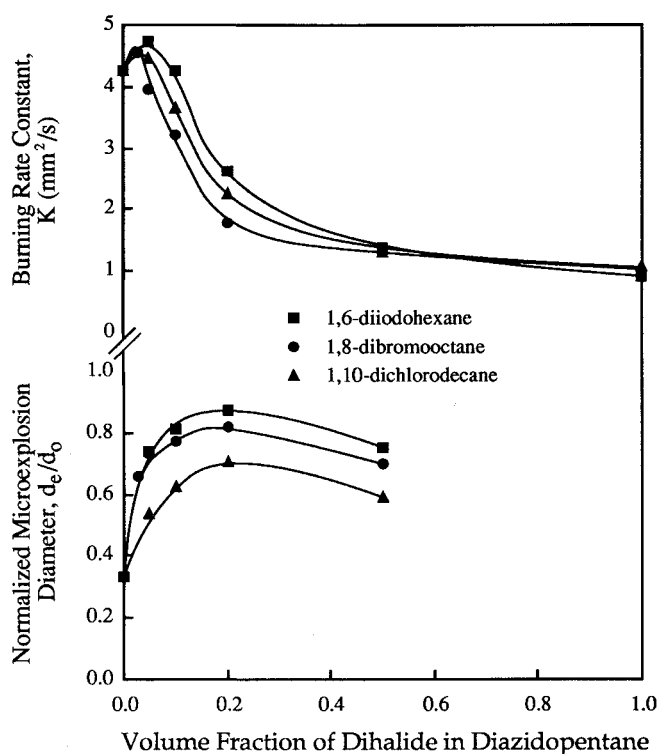
than that of diazidopentane, whereas the lower volatility dihalides are 1,10-dichlorodecane, 1,8-dibromooctane, and 1,6-diiodohexane, whose boiling points are approximately 60 K higher. No light diiodide was tested because only diiodomethane has sufficient volatility, but it differs too greatly in chemical structure and reactivity from the other compounds.

Results show that the gasification behavior of the different volatility mixtures exhibits the same general trend as that of the equal volatility mixtures. That is, for low levels of dihalide addition, the burning rate is slightly increased while the microexplosion event is significantly advanced. The facilitated microexplosion event leads to significantly enhanced overall gasification rates. For higher levels of dihalide addition and hence diazide dilution, the burning rate decreases fairly rapidly while the microexplosion event is also gradually retarded.

These results demonstrate the strong, beneficial, chemical influence with only a small amount of dihalide addition, regardless of its volatility differential in relation to the host dia-

zide, at least for the chemical compounds tested in the present study. The insensitivity of the droplet gasification rate on the volatility differential is also in agreement with the established understanding of the mechanism governing the gasification of multicomponent droplets. That is, previous experimental and theoretical multicomponent droplet studies[10,11] involving mixtures that do not react in the liquid phase have shown that the droplet gasification response is critically influenced by the relative rates of liquid-phase mass diffusion relative to droplet surface regression. For a droplet of conventional liquid fuels undergoing pure vaporization in an environment that is substantially cooler than that within typical combustors, the surface regresses sufficiently slowly such that enough time is available for mass diffusion to be effective throughout the droplet interior. Consequently, the relative rates with which components of different volatilities are gasified at the surface are strongly controlled by their volatility differentials. However, in the more realistic situations involving fairly rapid rates of gasification of droplets undergoing either pure vaporization in a hot environment or combustion, mass diffusion would exert greater influence by limiting the rates with which different components can be supplied to the droplet surface. The influence of volatility differentials is, therefore, correspondingly reduced. Since the present system of mixtures has very high gasification rates, it is reasonable that volatility differential effects are minimized.

Thus far, the experimental results have been restricted to an examination of a single test azide (1,5-diazidopentane) for simplicity. Additional results for dihalide blending with 1,4-diazidobutane are also presented to demonstrate the applicability of the current findings to other diazides. Figures 9 and 10 compare the burning-rate constants, the normalized microexplosion sizes, and the overall burning-rate constants of both diazides with the addition of the low-volatility diiodohexane. The results are qualitatively similar with those of the test diazide, diazidopentane, and the ability of the dihalide to enhance droplet microexplosion is again demonstrated.

Because chemical effects appear to be so important, it is of interest, then, to consider the influence of reaction stoichiometry. The experimental results show that, for all of the diazide/dihalide mixtures tested, the peak enhancement always occurs with 5–15% by volume of dihalide added to the diazide. In molar values, the mole ratio of the functional groups (halogen/azide) at the peak behavior lies within a fairly narrow concentration band and always is less than unity, typically with the halogen/azide mole ratio lying between 0.05–0.2. Since the extent of dihalide addition is small and the dihalide molecules themselves consist of relatively large numbers of C and H, which basically determine the fuel exothermicity, dihalide addition has minimal influence on the combustion properties[8] in terms of fuel exothermicity and hydrogen scavenging in terms of the H pool. Indeed, our experiments did
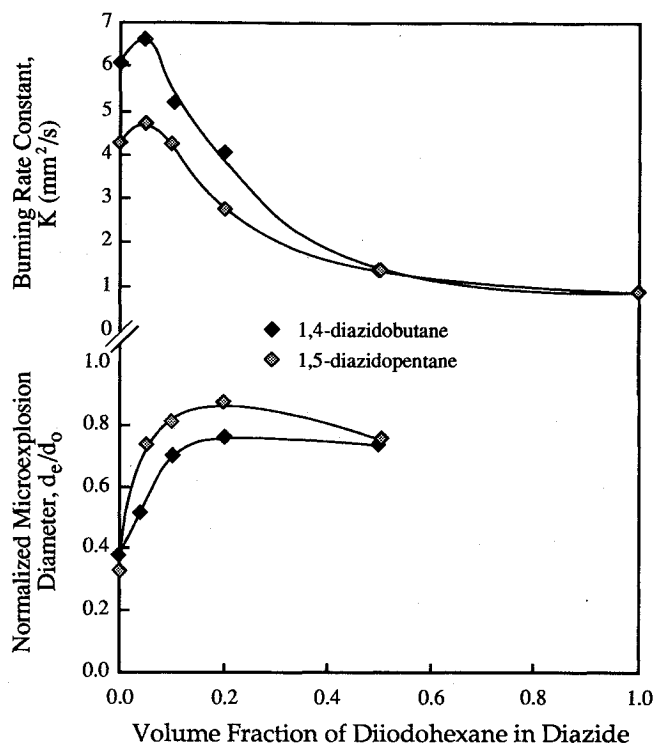
Fig. 9 Burning-rate constant K and normalized microexplosion diameter for mixtures of 1,4-diazidobutane and 1,5-diazidopentane with various amounts of 1,6-diiodohexane, at 1 atm.
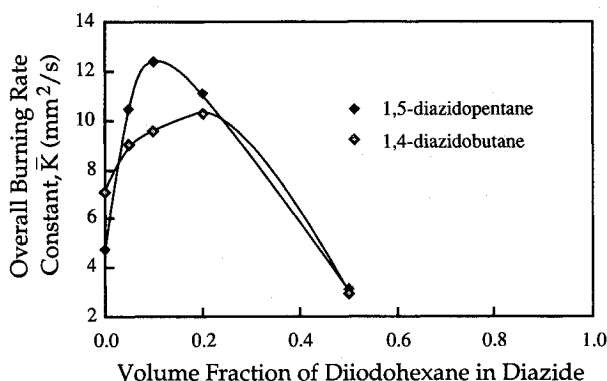

Fig. 10 Overall burning-rate constant $\bar{K}$ for mixtures of 1,4-diazidobutane and 1,5-diazidopentane with various amounts of 1,6-diiodohexane, at 1 atm.

not observe any decrease in the combustibility of the fuel mixture. Finally, it is also significant to note that enhanced burning is only observed for mixtures of diazide and dihalide. Experiments where the dihalide or diazide is replaced by twice as much monohalide or monoazide, of the same volatility, do not show the same enhanced behavior.

Pure Diazide Vaporization Under Elevated Pressures

The gasification behavior of diazides and diazide/dihalide mixtures have already been demonstrated to possess significantly enhanced burning and microexplosion characteristics as compared to conventional hydrocarbon fuels under ambient pressures. Because these materials have potential applications in pressurized environments, it is of interest to examine their behavior under elevated pressure. Fundamentally, since the droplet temperature should increase with increasing pressure because of the corresponding elevation of the liquid boiling point, and since liquid-phase reactions should also be sensi-
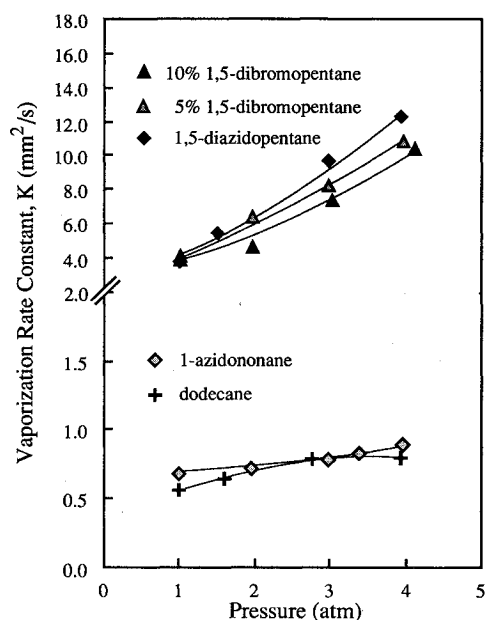

Fig. 11 Vaporization-rate constant K as function of pressure for decane, 1-azidononane, 1,5-diazidopentane, and mixtures of 1,5-diazidopentane with 5 and 10 volume % of 1,5-dibromopentane.
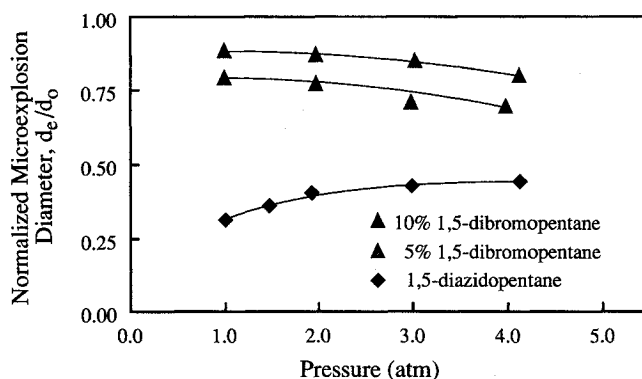

Fig. 12 Normalized microexplosion diameter as function of pressure for 1,5-diazidopentane and mixtures of 1,5-diazidopentane with 5 and 10 volume % of 1,5-dibromopentane.

tively affected by the droplet temperature, increases in the system pressure could lead to further enhancements in the droplet response.

Preliminary combustion experiments at elevated pressures determined that the diazide flame is quite luminous, hence making photographic imaging of the droplet size difficult. Thus, the pressurized experiments have been performed in heated, inert environments. The compounds chosen for comparative studies are 1,5-diazidopentane, 1-azidononane, and dodecane, which all have nearly identical boiling points and latent heats. The pressure range of investigation is from 1 to 4 atm.

The vaporization rates of the monoazide and diazide are presented in Fig. 11 at various pressures. These results differ slightly from the vaporization rates shown in Fig. 1 because of the somewhat higher chamber temperature. The vaporization rates, however, still follow the same general trends and do not impact on any of the present observations or conclusions. The results show that the vaporization rate of diazidopentane is remarkably sensitive to pressure, rising from about 4 mm^2/s at 1 atm to 12 mm^2/s at 4 atm, representing a threefold increase. As a control, an equal-volatility monoazide (azidononane) and an equal-volatility hydrocarbon (dodecane) have also been examined under identical conditions. The comparative results are superimposed in Fig. 11 and confirm that over the 4-atm pressure range, the vaporization rate is increased
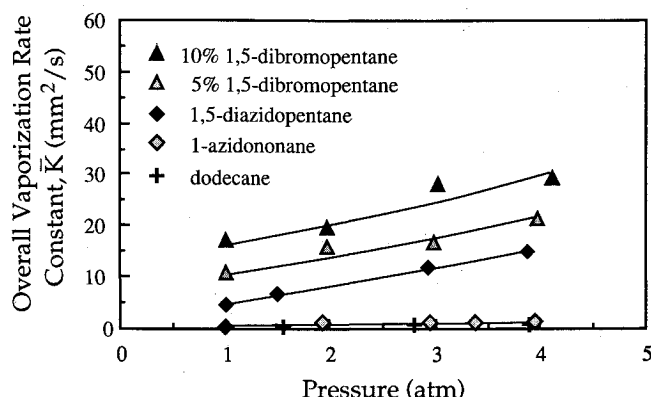
Fig. 13 Overall vaporization-rate constant $\bar{K}$ as function of pressure for decane, 1-azidononane, 1,5-diazidopentane, and mixtures of 1,5-diazidopentane with 5 and 10 volume % of 1,5-dibromopentane.

only about 20%. Thus, alkanes and monoazides behave similarly, as previously observed in atmospheric pressure experiments. The fact that the diazide gasifies so much more rapidly than the monoazide is again clear evidence of the role of liquid-phase kinetics and exothermic decomposition on the gasification of the more densely substituted diazide. Thus, as expected, an increase in the droplet temperature due to an increase in the system pressure, and thereby the liquid boiling point, can induce an exponential increase in the heat release rate within the diazide droplet and, consequently, the dramatic increase in its gasification rate.

Figure 12 presents the normalized microexplosion diameter for diazidopentane at various pressures and shows that, whereas the vaporization rate of the diazide is substantially increased at higher pressures, the microexplosion event is only moderately advanced.

Diazide/Dihalide Mixture Vaporization Under Elevated Pressures

Recognizing that dihalide addition and pressure elevation can enhance droplet microexplosion and its gasification rate, respectively, their coupled effects are studied by using mixtures of diazidopentane and dibromopentane under elevated pressures. The data, presented in Figs. 11 and 12, show that dihalide addition does not further improve the vaporization-rate response with increasing pressure. The state of microexplosion, however, is significantly advanced with dihalide addition at all pressures. For a given amount of dihalide addition, increasing pressure only moderately advances the state of microexplosion.

Figure 13 shows the overall vaporization-rate constants determined from the data of Figs. 11 and 12. Compared with the 0.5–1-mm²/s gasification rates of the alkanes and monoazides, the combined effects of diazido functionality, dihalide addition, and pressure elevation in boosting the overall vaporization rates of the diazide fuel to values approaching 30 mm²/s are quite impressive.

Concluding Remarks

In this investigation we have successfully achieved our objectives of improving the droplet gasification response of diazido fuels both through the judicious use of chemical additives in the form of alkyl dihalides, primarily to promote microexplosion, and by using elevated pressures to increase the droplet

temperature and thereby intensify the liquid-phase decomposition reactions that drive the gasification of these materials. The alkyl dihalides investigated have the same terminally disubstituted form as the diazides. Although dihalide addition minimally affects the gasification rate, the addition of about only 5–10 volume percent dihalide significantly advances droplet microexplosion and thereby greatly reduces the droplet lifetime. Whereas the results are obviously composition dependent, similar results are not obtained for twice as much monohalide or monoazide. These general results are observed for all of the dihalides investigated, regardless of their volatilities or specific halogen atoms. The specific reaction mechanism through which the beneficial effects of dihalide addition are realized, however, has not been identified.

Droplet vaporization experiments performed in nonoxidizing environments at elevated pressures have conclusively demonstrated that, whereas the state of microexplosion is only minimally affected, the gasification rate is significantly enhanced at higher pressures. This large gasification rate increase is not observed for monoazides or hydrocarbons of equal volatility and, therefore, again delineates the importance of the liquid-phase reactions that are expected to be highly temperature sensitive. Finally, the study also demonstrates that the individual effects of dihalide addition and pressure elevation can be coupled to maximize the overall gasification rate enhancement.

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