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Characterization of Soot Production by Synthetic Fuels

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Abstract: This study presents the sooting tendencies exhibited by binary mixtures of synthetic fuels i.e. n-heptane/toluene and isooctane/toluene, in terms of Yield Sooting Indices (YSIs). These synthetic fuels are indeed incorporated into almost every gasoline surrogate fuel used for experimental study or performing numerical simulation of combustion process in engines. Diffusion flames of methane diluted by nitrogen and doped with the vapors of the aforementioned mixtures are established over the Santoro's burner. Two-dimensional maps of soot volume fraction in these flames are then inferred from line-of-sight attenuation measurements. The measured soot volume fractions are converted into apparatus-independent YSIs which affords the possibility to compare the relative influence of fuel formulation on soot production. The trends in soot production capacity as a function of toluene mole fraction are then analyzed by curve fitting YSI data. A non-linear evolution of the sooting tendency as a function of toluene mole fraction in the binary mixtures reveals especially for isooctane/toluene blends at lower toluene mole fraction. Two Quadratic equations adequately model the evolution of YSI as a function of toluene mole fraction in n-heptane/toluene and isooctane/toluene blends. The trends as inferred from these equations and experimental results are in fair agreement with earlier studies, which further sustain the consistency of the YSI methodology. Interestingly, in an earlier study[Combust. Flame 161 (2012) 1575-1586], almost linear evolution of sooting tendencies has been evidenced for the blends of n-heptane and isooctane. This clearly points towards the presence of a synergistic effect only visible when certain types of hydrocarbons are present in the fuel mixture (branched-chain alkanes and alkyl benzenes in our case). The quadratic equations presented here can be very helpful while formulating fuel mixing strategies in view of the future engine and environmental exigencies and can provide preliminary predictions of soot emissions characteristics of a fuel.

Keywords: Alternate/Synthetic Fuels, Soot Production, Yield Sooting Index

1. INTRODUCTION

Internal combustion engines are a major source of locomotion and power generation but the combustion process in both gasoline and diesel engine leads to the formation of number of pollutants e.g. Carbon monoxide (CO), nitric oxide (NO) and nitrogen oxide (NO_2) collectively called as NO_x, unburnt hydrocarbon (UHC) and Particulate matter. These pollutants are ultimately emitted into the atmosphere where they pose a serious health hazard to human health. Many recent studies have hinted toward a strong link between high mortality rates to increased concentration of fine and ultra-fine particles in city atmosphere (Patricia *et al.*, 2012). (OŚhaughnessy, 2012).

Soot formation during combustion is the result of complex and competing physico-chemical processes which lead to the inception, growth and subsequent oxidation of soot particles from the species formed during the pyrolysis of a complex blend of hydrocarbons in fuel (D'Anna2009). The recent development in computational power now affords the possibility to develop comprehensive methodologies to refine the understanding of soot formation pathways by developing numerical models and testing them against a set of experimental measurements performed on laboratory reference flames (Liu, *et al.*, 2001). This strategy require sophisticated computational and experimental tools capable of providing accurate and meaningful data at reduced cost and time.

All practical fuels are liquid mixtures of hundreds of hydrocarbons at atmospheric conditions (Pitz *et al.*, 2007) "Every chemical compound present in fuel mixture has a variable and coupled effect on combustion chemistry engine performance, regulated engine emissions and especially soot production (Ußner *et al.*, 2009) Many works now employ surrogate fuels to minimize the computational cost and time needed to develop a molecular-level understanding of fuel combustion process. Dedicated efforts are being made to develop optimized surrogate fuels for gasoline, diesel and jet engines based on available reaction kinetics data and experimental validation results (Pitz *et al.*, 2007). The cumulative effort has resulted in the development of blends which are mixtures of at least n-heptane, isooctane and toluene (Pitz *et al.*, 2007) and have shown to be capable to reproduce quite adequately the various aspects of combustion behavior of gasoline.

To compare the sooting tendencies of a large number of chemical compounds, surrogate fuels, commercial fuels and mixture of hydrocarbons and bio-fuels, different methods like smoke point height, threshold sooting index (TSI) and Yield sooting Index

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measured in laminar diffusion flames have been reported (Dotson, *et al.*, 2011) (Calcote and Manos, 1983) (McEnally and Pfefferle, 2007) (McEnally and Pfefferle, 2011) Among these methodologies, YSI methodology has shown to be more reliable with uncertainties which are lower as compared to the ones normally associated with TSI methodology. In the present study, the experimental procedure to reproduce the methodology recommended by McEnally and Pfefferle is used to measure the sooting tendencies of binary mixtures of n-heptane, isooctane and toluene. The influence of the blending is then assessed for binary mixtures of n-heptane/toluene and isooctane/toluene.

2. MATERIAL AND METHOD

The methodology detailed by McEnally and Pfefferle (McEnally and Pfefferle, 2007) and extended and assessed for consistency at higher doping concentrations by (kashif *et al*, .2014) is reproduced to measure soot production capacity of binary mixtures. The experimental setup consisting of axis-symmetric coflow burner, fuel metering, fuel evaporation and mixing, and optical diagnostics has been carefully explained in (kashif *et al.*2014). A schematic of fuel metering, evaporation and mixing diagram is shown in **(Fig. 1).**

The burner used in this study is a Santoro type burner. The axis of symmetry is (Oz) with its origin located at the burner tip, defining the height above the burner (HAB). The cross-stream coordinate is r, which is the distance from the axis of symmetry. The reactants came from high-purity gas cylinders (CH_4; 99.9 % stated purities), reagent-grade bottles (n-heptane and isooctane and toluene; 99.5 %), and a compressor (air). In summary, the method suggested by McEnally and Pfefferle provide the apparatus-independent Yield Sooting Index (YSI) of a fuel from the maximum soot volume fraction,$f_{v,max}$= max $\{f_v(\boldsymbol{r,z_{meas.}})|r \in [\boldsymbol{0,\ R}]\}$ measured in the doped methane flame at a fixed height $\boldsymbol{z_{meas}}$ above the burner using Eq.(1):

$$\boldsymbol{YSI = Cf_{v,max} + D} \quad \textbf{(1)}$$

Here, ***C*** and ***D*** are apparatus-specific parameters that are determined by the arbitrary ***YSI*** values attributed to two reference fuels. For present study YSI values of Benzene and toluene reported by McEnally and Pfefferle (McEnally and Pfefferle,2011) .

2.1. Mixture preparation

The doping concentration and composition of carrier gas is different from the one reported in an earlier study on primary reference fuels. Therefore, the YSI values are not directly comparable with the one reported in current article but the effects of fuel blending on YSI can be compared qualitatively especially for synergistic effects reported later in the article.

As depicted in the **(Fig. 1)**, the liquid fuel whose sooting tendency is to be quantified is stored in a specific tank and is pushed by an inert gas, i.e. Argon, and flows through a Coriolis mass flow controller. The fuel is then vaporized and mixed with a carrier gas, i.e. methane, using Bronkhorst controlled evaporation and mixing (CEM) system. The gaseous mixture is carried via a heated line to the inner central duct of the burner. As resumed in (kashif *et al.* 2014).the flame once stabilized is probed by LEM to get soot volume fraction necessary to calculate yield sooting index of the fuel mixture in the doped methane diffusion flames.

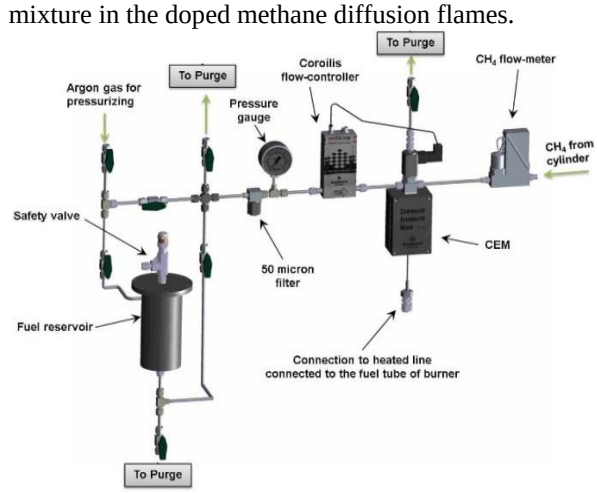

Fig.1:Experimental arrangement for soot volume fraction measurements in doped methane flames. Various components of liquid metering and doping system are visible.

The experimental parameters that were constant during the whole experimental campaign are given in **(Table. 1).** In brief, we investigated dopant concentrations corresponding to two reference benzene mass flow rates, i.e. **0.42*mg/sec*** (**1.5*g/hr***) and **0.56*mg/sec***(**2.0*g/hr***), at room temperature. These flow rates correspond to the vapor mole fractions of $\mathbf{X_{vap,1} = 1.86 \times 10^{-2}}$and $\mathbf{X_{vap,2} = 2.47 \times 10^{-2}}$, respectively. The mass flow rate fora given liquid fuel to be investigated is then adjusted such that its vapor mole fraction is equal to $\mathbf{X_{vap,\,i}}$ **(i = 1,2)**; calculated with$\pm$**0.1**% relative uncertainty from compiled liquid phase densities and species molecular weights *CRC Handbook of Chemistry and Physics, 94th Edition*, (2004).

2.2. Experimental and post-processing procedure

The details of the methods to calculate the YSI of a given fuel from Eq.(1), suggesting different alternatives to the evaluation of $\boldsymbol{f_{v,max}}$are reproduced here and have been explained in detail in (kashif *et al.* 2014).

1. The first method -an extension of the original definition of McEnally and Pfefferle (McEnally and Pfefferle,2011) is referred as $\boldsymbol{YSI_{Max.Ax.fv}}$ and is based

on the maximum soot volume fraction on the axis of the flame (but not at a fixed location).

2. The second method is identified by $YSI_{GlobalMax.fv}$ based on peak soot volume fraction $f_{v,max}$ into the whole distribution.

3. The third method is referred as $YSI_{Max.Int.fv}$. Here, the integrated soot volume fraction $F_v(z)$was first computed as follows:

$$F_v(z) = 1/(\pi R^2) \int_0^R 2\pi f_v(r,z)dr \qquad (2)$$

Then the maximum value of $F_v(z)$was used to evaluate YSI. $F_v(z)$is an evaluation of the actual soot load at a given HAB into the flame.

For every flame investigated, 400 consecutive fields of raw integrated extinction coefficient are first processed from a 20 s long LEM recording. Then a deconvolution procedure based on the onion peeling method stabilized using Tikhonov regularization (Daun, *et al.*, 2006) is applied to every field, providing 400 maps of local extinction coefficient $\kappa_\lambda^{ext}(r,z)$. The local soot volume fraction is finally inferred from the local extinction coefficient using the following equation:

$$f_v(r,z) = \lambda\kappa_\lambda^{ext}(r,z)/(6\pi(1+\alpha_{sa})E(m)) \qquad (3)$$

Where $E(m)$is a function of the complex refractive index m of soot. α_{sa} is the relative contribution of scattering to extinction, therefore is directly related to soot morphology. Three different maximum measured soot volume fractions, axial, global and integrated are extracted from the soot maps in doped flame for all fuel blends.

The morphology of primary soot particles and aggregates depends strongly on liquid fuel chemistry which results in a significant discrepancy of α_{sa} that can over-estimate the value of $f_{v,max}$ for benzene and toluene and binary mixtures of toluene with heptane and isooctane. The value of α_{sa} were adjusted following the procedure outlined in (kashif *et al.*2014). However, the difference in the YSIs calculated -with and without taking account the relative contribution of α_{sa}- are of the order of experimental uncertainty. Thus, in the following, the denominator in Eq.(3) is set to 5.3 for n-heptane, and isooctane (α_{sa}), and to 5.8 for Benzene and toluene (α_{sa}= **0.1**). For binary mixtures of n-heptane/toluene and isooctane/toluene an interpolated value of α_{sa} based on the molar ratio of the blend is used to take into account increasing residence time and varying soot morphology which is influenced predominately by increasing mole fraction of toluene.

Table 1: Experimental Parameters kept constant during the evaluation of YSI.

Parameter	Present study	McEnally and Pfefferle (McEnally, *et al.*, 2011)
Ambient Pressure (atm)	1.0	1.0
Ambient Temperature (°C)	20	20
Carrier gas Composition	CH_4(50 %) N_2(50 %)	CH_4(55 %), N_2(45 %)
Carrier gas flow rate (cm^3/min)	400± 2.4	605
Coflowing air flow rate (cm^3/min)	60000 ± 360	30000
Evaporator temperature (°C)	150 ±1	N.A.
Heated line temperature (°C)	150 ±2	145
Reference YSIs	YSI(Benzene)=30	YSI(Benzene)=30
	YSI(toluene)=43.5	YSI(dihydronaphthalene)=100

3. RESULTS AND DISCUSSION

The synergistic effect of blending on the evolution of sooting propensity (measured here as YSI) has been revealed especially for binary mixtures containing isooctane and toluene. This trend is in agreement with the available literature (Choi, *et al.*, 2011). On the other hand blends of n-heptane and toluene show linear evolution of YSI with increasing mole fraction of toluene. But for low mole fractions of toluene the sooting propensity of binary mixtures of n-heptane/toluene might be greater than that predicted by linear interpolation. This clearly shows the effect of chemical structure of a compound on the sooting tendency are different for a liquid fuel blend especially in the presence of aromatic hydrocarbons.

3.1. Effect of blending on sooting propensity

(Fig. 2) exhibits for different mole fractions of toluene the soot volume fraction fields inside the methane flames doped with vapors of different blends ofn-heptane/toluene and isooctane/toluene ($X_{vap,2}$=2.47 10^{-2}). The soot maps in flames doped with benzene are shown in the extreme right column. These fields show the global sooting behaviors of n-heptane, isooctane, toluene, benzene, and binary mixtures of n-heptane/toluene and isooctane/toluene in coflowing diffusion flames established over modified Santoro type

burner. The sooting tendency increases monotonically with increasing mole fraction of toluene in the dopant liquid mixture both for heptane or isooctane, which is qualitatively in accordance with the available data (Choi, *et al.*, 2011).

To further elucidate the effect of blending on soot formation for binary mixture of n-heptane/toluene and isooctane/toluene, the evolution of the integrated soot volume fraction $F_v(z)$into the doped flames of methane is plotted against height above burner (HAB) in **(Fig. 3).** The first trend which is also visible qualitatively in **(Fig. 2)** is the zone of appearance of first soot particles which shifts gradually upstream to lower heights in the flame with increasing toluene mole fraction. The peak level increases monotonically with the increasing amount of toluene into these blends while the peak shifts towards lower HAB. Moreover, the higher slopes of the increasing parts of the curves indicates an increasing soot formation rate with increasing toluene mole fraction.

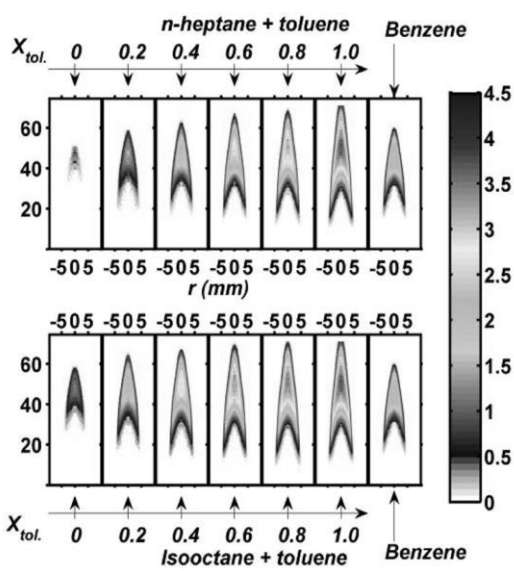

Fig.2: Soot volume fraction fields in methane diffusion flames doped with different blends of n-heptane and toluene and isooctane and toluene. $X_{tol.}$here represents the mole fraction of toluene in liquid fuel mixture. The vapor mole fraction in the fuel stream is $X_{vap,2}$ = 2.47 x 10^{-2}.

In **(Fig. 3)** the synergistic effect of isooctane/toluene blending on integrating soot volume fraction is clearly visible. The blends with lower toluene mole fractions show an augmented soot production capacity while at higher toluene mole fraction the evolution of integrated soot volume fraction approach more closely in magnitude and shape to the one representing pure toluene doped flames. The curves representing binary mixture of toluene with heptane approach steadily and quite linearly to that of pure toluene while a non-linear evolution of integrated soot volume fraction is visible for the curves depicting blends of isooctane and toluene.

(Choi, *et al.*, 2011) resumed the important pathways leading to the formation of poly-aromatic hydrocarbons (PAHs) which are precursors to soot. The dominant reaction which leads to the formation of benzene (**A1**) and naphthalene from toluene are: **$C_6H_5CH_3$ + H → A1(C_6H_6) + CH_3; A1 -CH_2 + C_3H_3→ A2($C_{10}H_8$) + H_2 and A1 - C_6H_5 + C_4H_4→ A2($C_{10}H_8$) + H; and A1 - C_6H_5 + C_4H_4→ A2($C_{10}H_8$) + H**

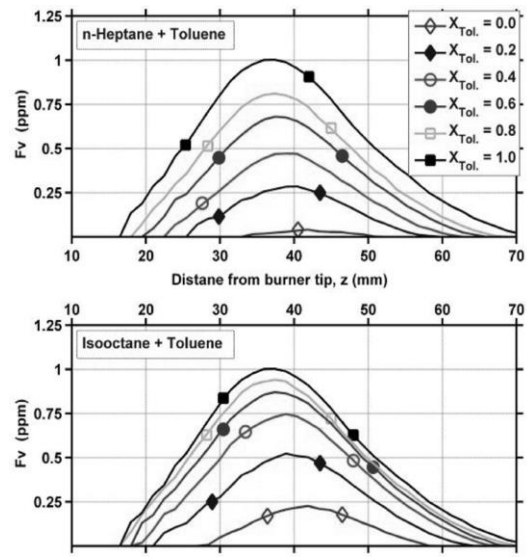

Fig.3: Evolution of the integrated soot volume fraction $F_v(z)$into methane flames doped with different blends of n-heptane/toluene and isooctane/toluene. The vapor mole fraction in the fuel stream is $X_{vap,1}$ = 2.47 10^{-2}.

In case of isooctane/toluene blends the higher concentrations of propargyl**H, C_3H_3** and vinylacetylene (**C_4H_4**) will accelerate naphthalene formation and its growth to heavier PAHs. This phenomenon reduces with the increase in toluene mole fraction and also is more dominant in case of isooctane/toluene blends because of the pyrolysis of heptane forms species which have little effect on PAH formation pathways. This synergistic behavior can be of great consequence as most of the surrogate fuels used in numerical simulations and engine testing contain toluene mole fraction in the range where this effect is not negligible. Hence, a more prudent approach is necessary while formulating a surrogate fuel matching the sooting tendencies of commercial gasoline.

3.2. Curve fit models for YSI($X_{tol.}$) for blends n-heptane/toluene and isooctane/toluene

(Fig. 4) show the Yield Sooting Indices exhibited by the different binary mixtures of heptane/toluene and isooctane/toluene studied at both vapor mole fractions.

A model scaling the absolute **YSI** as a function of $X_{tol.}$ is likely to be relevant in order to predict non-linear evolution of YSI with X_{tol}. To this end, the second-order polynomial in Eq.(4) is expected to decently model the non-linearity that can be observed in Fig.4:

$$YSI = a(X_{tol.})^2 + b(X_{tol.}) + c \qquad (4)$$

The coefficients are reported in Tab.2. The detail explanation of the curve-fitting procedure and its implementation in MatLab environment can be found in (Coleman 1996)and (Kenney and Keeping, 1962).Consequently, Fig.4 exhibits for both $X_{vap.}$ the experimental values of **YSI** calculated using three different methods defined earlier as a function of $X_{tol.}$for binary mixtures of n-heptane and isooctane with toluene. All mean experimental YSIs are indicated by symbols. The red and green bands represent the 95% confidence intervals for blends of n-heptane/toluene and isooctane/toluene evaluated using Eq.(5):

$$CI(X_{tol.}) = \overline{YSI}(X_{tol.}) \pm 1.96 \times \sigma_{max}) \qquad (5)$$

where YSI is the average value of the YSIs measured at a given set of conditions ($X_{vap,i}$, $X_{tol.}$) and σ_{max} is the maximum value among the standard deviations computed at every set of conditions ($X_{vap,i}$, $X_{tol.}$) investigated. The interval ±1.96 × σ_{max} represents 95% of the area under the standard distribution curve (Kenney, and keeping 1962).

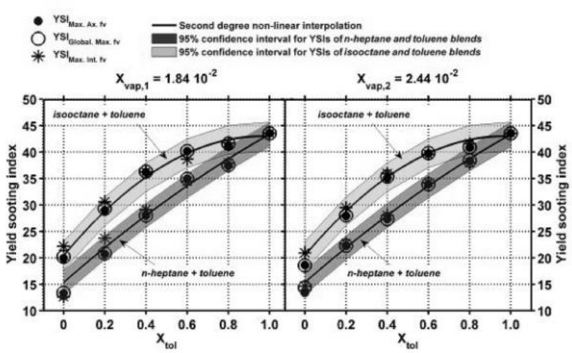

Fig.4: Yield Sooting Indices of binary mixtures of n-heptane and toluene and isooctane and toluene, from pure n-heptane or isooctane ($X_{tol.}$=0) to pure toluene ($X_{tol.}$=1).

Table 2: Coefficients of the polynomial fitting the experimental YSIs (see Eq.(4)).

Coefficients	heptane/toluene	isooctane/toluene
a	**-6.07**	**-24.30**
b	**34.05**	**46.82**
c	**15.34**	**20.40**

The magnitude and sign of the ratio of coefficients ***a***and***b*** demonstrate quantitatively the non-linear trends of YSI as a function of toluene mole fraction. The ratio of the coefficients of the quadratic equation modeling YSI as a function of $X_{tol.}$shows that while YSIs for the binary mixtures of n-heptane/toluene evolve almost linearly as function of toluene mole fraction $((a/b)_{heptane}$ =-0.178), this trend in far from linear for isooctane/toluene blends ($(a/b)_{isooctane}$ =-0.51).This clearly shows that the parabolic term in the YSI model Eq. 4 in case of isooctane/toluene blends is important, hinting at a non-linear evolution which dampens YSI at higher toluene mole fraction.

(Fig. 5) shows the triangular surface plot of the interpolated YSI values for tri-component blends of n-heptane, isooctane and toluene. The predicted YSI values of tri-component blends are found by interpolating the YSI values of binary blends reported above. The mole fraction of one of three component is 0 along any of three axis e.g. the mole fraction of toluene along isooctane is zero, hence the $X_{tol.}$= 0 for any point lying on this axis for a blend of n-heptane and isooctane and so on.

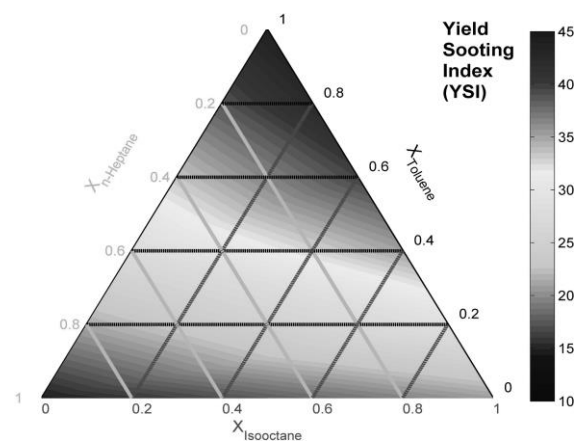

Fig.5: Tri-component surface plot representing YSI values of n-heptane, isooctane and toluene (at the vertices) and binary blends (along the edges), and interpolated YSI values from tri-component blends (area enclosed inside the triangle).

The sooting propensity of any blend can be readily found on the surface plot by locating the points corresponding to the mole fractions of n-heptane, isooctane and toluene on their respective axes. Then lines are drawn from each axis following the iso-concentration (at 45***degree*** to the respective axis) for that particular components (the iso-concentration lines and axis label for a compound are of the same color i.e. black for toluene, green for n-heptane and magenta for isooctane).

The YSI value at the point of intersection of three iso-concentration lines is the predicted YSI value for the tri-component synthetic fuel. Hence, a quick comparison of the soot production capacity can be

readily made by projecting the points onto the surface plot in Fig. 5. Another interesting aspect of the graph is the bands of iso-YSI values evident by same colored zones on the surface plots. This clearly shows the existence of a number of different tri-component blends having the same soot production capacity. Therefore, other criteria of selection like auto-ignition behavior, ignition delay, octane rating, laminar burning velocity and strain extinction rate etc. need to be included in the selection criteria for a surrogate or alternative fuels.

4. CONCLUSIONS

The sooting propensities of binary mixtures of n-heptane/toluene and isooctane/toluene were measured in terms of Yield Sooting Indices (YSI) in doped methane diffusion flames. A synergistic effect on soot production tendencies is quantified in terms of measured YSI and an equation modeling YSI as function of toluene mole fraction for these binary mixtures is fitted to the experimental values. A quadratic equation of the parabolic curve fitted to the measured values of YSI has been modeled for binary mixtures of n-heptane/toluene and isooctane/toluene. The modeled YSI equations provide YSI values of a binary mixture of n-heptane or isooctane with toluene with an uncertainty of **±2.5 YSI** units. The results presented highlights the non-linear effect on soot production capacity by fuels containing branched-chain and aromatic hydrocarbons. The presence of strong synergistic effect on sooting propensity of binary mixtures necessitates a detail study of fuel formulation policies in view of the evolving emission regulations. A surface plot showing interpolated YSI values of tri-component blends of n-heptane, isooctane and toluene is also presented which can be used to get an early prediction of sooting propensities.

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