

Combustion Characterization of Solvents used in Coil Coating Processes: Experiments and Kinetic Modelling

Z. Malliotakis¹, P. Weinbrecht², F. Cameron³, D. Katsourinis^{*1}, G. Vourliotakis¹, C. Weis², G. Skevis⁴, D. Trimis², M. Founti¹

¹Laboratory of Heterogeneous Systems & Combustion Systems, School of Mechanical Engineering, National Technical University of Athens, Athens, Greece.

²Engler-Bunte-Institute Combustion Technology, Karlsruhe Institute of Technology, Karlsruhe, Germany.

³Institute for combustion technology, RWTH Aachen University, Aachen, Germany.

⁴Chemical Process and Energy Resources Institute, Centre for Research & Technology Hellas, Thessaloniki, Greece.

Abstract

A combined experimental and chemical kinetics modelling approach is presented to account for the combustion behaviour of solvents utilized in coil coating processes. Heating values and laminar burning velocities of typical industrial solvent formulations comprising alcohols, ethers, esters and aromatics are experimentally investigated. Due to the complexity of species participating in the solvent formulations surrogate solvents are introduced, one for each considered formulation. An “in-house” chemical kinetics mechanism has been extended in order to take into account the solvents’ combustion and consists of 321 species participating in 1826 reactions. Its overall performance is validated against the laminar burning velocity measurements. A good qualitative and quantitative reproduction of the experimental curves is depicted with maximum discrepancies observed in the range of 10-15%.

Introduction

The present study can be classified under the framework of introducing alternative, non-traditional fuels to industrial applications in order to enhance their sustainability, increase their efficiency and reduce their environmental impact. Coil coating is an advanced finishing technology available for different metal substrates, specifically steel and aluminum, with several millions of square meters processed each year [1]. It is a continuous industrial process used to efficiently coat metals with organic coatings [2] through a curing process. The produced coated metal coil can be subsequently used for numerous applications such as roofing, water management systems, etc [2,3]. The paints, which have to be applied on the metal sheets consist of resins, pigments, crosslinkers and solvents: the latter are essentially complex mixtures of various organic compounds [4]. A typical coil coating process utilizes high temperature process to reach a peak metal temperature (PMT) of 230–240 °C to allow for the full curing to be completed [2]. Conventional methods for curing rely on convective heat transfer using hot air generated by combustion. A novel concept proposes to use radiative heat transfer emitted from porous radiant burners and utilize the solvents as fuel for process heat generation [5].

In order to assess the potential of using alternative liquid fuels with complex compositions a comprehensive understanding of their combustion properties is required. Furthermore, surrogate fuels are introduced to facilitate and reproduce the kinetic modelling of the alternative fuels combustion. This approach has been extensively reported in transportation [6–8].

Laminar burning velocity is a key parameter for reacting mixtures containing fundamental information

regarding reactivity, diffusivity and exothermicity and helping in the description of various combustion phenomena (e.g flame stability, flashback, blowout etc) [9]. Furthermore it is widely used for the validation of detailed chemical kinetic mechanisms [6,10,11].

This work presents a combined experimental and kinetics modelling approach for the characterization of different solvent formulations applied in coil coatings. Higher heating value and laminar burning velocity were measured using appropriate techniques. The measurements were subsequently used: (a) for the development of surrogate mixtures and (b) for the validation of a detailed kinetics mechanism extended to account for the surrogate mixtures combustion.

Characterization of Solvents

Four different solvent formulations are examined. The basic chemical families identified in the four formulations compositions as well as their respective concentration ranges (% wt) are presented in Table 1. As it can be seen the solvents comprise mostly aromatics (42-62%), esters (10-52%) and ethers (5-30%). Alcohols (1-15%) can be found in lesser quantities in two formulations. It should be noted that due to the complexity of the mixtures a detailed presentation of the solvent compositions is not feasible. However, an indicative list of the main species identified per formulation is depicted in Table 2.

Table 1. Concentrations (% wt) ranges of chemical families identified in the solvents’ compositions.

Solvent	Aromatics	Esters	Ethers	Alcohols
Form. 1	42-52	42-52	-	-
Form. 2	40-50	33-43	5-15	5-15
Form. 3	47-57	15-25	20-30	-

Table 2. Chemical species identified in the solvent formulations.

Chemical families	Indicative Chemical Species
Aromatics	Solvent naphtha, light aromatics, heavy aromatics, xylene
Esters	<ul style="list-style-type: none"> • dibasic ester, ethyl acetate, • butylglycol acetate, 1-methoxy-2- • propanol acetate
Ethers	<ul style="list-style-type: none"> • dipropylene glycol ether, butyl glycol, • butyl diglycol ether
Alcohols	<ul style="list-style-type: none"> - butanol, iso – butyl alcohol, 2- - toxyethanol.

Heating Value Measurements

The solvent formulations’ heating values were measured with the use of an oxygen bomb calorimeter. (Parr 6220 from Parr Instruments). Solvent samples were handled in stainless steel capsules. The capsules containing the solvent sample were sealed with a tape free of chlorine and as low in sulfur as possible. The tape recommended by Parr (Part No. 517A) was used. Initially the tape heat of combustion was determined by running five blank tests with tape alone using samples weighing approximately 1 gr and results indicated a mean value of 6053 cal/gr. Samples were measured according to the following process: initially the empty capsule was weighed and subsequently the top of the capsule was covered with tape. The solvent sample was added with a syringe in order to mass losses due to evaporation of the most volatile components in the solvents composition and the filled cup was re – weighed. The capsule was set in the holder and the auxiliary fuse was adjusted in order to touch the center of the tape disk. After closing the lid, the calorimeter bomb was filled with oxygen at a pressure of approximately 30 bar for 1 min. The filled vessel was then placed into the calorimeter and filled up with 2 lt of water. The initial temperature of the water was approximately 27 °C. The samples weight varied from 0.4 g to 0.9 g with an accuracy of 0.1 mg. The errors of each set were calculated from the scattering of the measured values.

Measured heating values are depicted in Fig.1 and range between 34-38 MJ/kg for all tested solvent formulations with standard deviations up to 0.3%. Additionally the heating value by means of the C, H, and O content of the fuel was calculated. The contents were determined via an external measurement at ASG Analytik-Service GmbH, Germany. For the calculation of the lower heating value H_L an equation for fuels with high oxygen content from Boie [12] is suitable and is composed as follows:

$$H_L = (8400 \cdot [C] + 22500 \cdot [H] + 1500 \cdot [N] - 2500 \cdot [O]) \cdot 4,184 \frac{kcal}{kg} \quad (1)$$

To calculate the higher heating value the evaporation enthalpy of water was added, (2)

$$H_H = H_L + 2257 \frac{kJ}{kg} \cdot m_W = H_L + 2257 \frac{kJ}{kg} \cdot [H] \cdot \left(1 + 0,5 \cdot 16 \frac{kgO}{kgH}\right)$$

Measured data and calculations are within the errors for all solvent formulations except from Formulation 4.

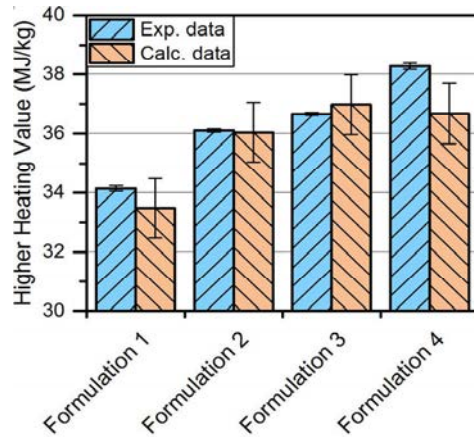


Fig. 1. Heating value measurements and calculations

Laminar Burning Velocity Measurement

The laminar burning velocity S_L of premixed solvent-air mixtures is determined according to the Heat-Flux-Method as introduced by de Goey et al. [13]. The method bases on the stabilization of premixed quasi-adiabatic flat flame downstream of a perforated plate. Assuming a flat flame the flow velocity corresponding to a nullified net heat flux of the plate represents the adiabatic burning velocity [14].

Fig. 2 shows the schematic sketch of the flat flame burner test rig. The key component is the burner head with the perforated burner plate. The perforated plate has a thickness of 2 mm, 50 mm diameter of perforated area with hole diameter of 0.5 mm. The temperature distribution inside the burner plate, in radial direction, is determined with ten type-E thermocouples (T3-T12) staggered along the radius in centered spiral arrangement.

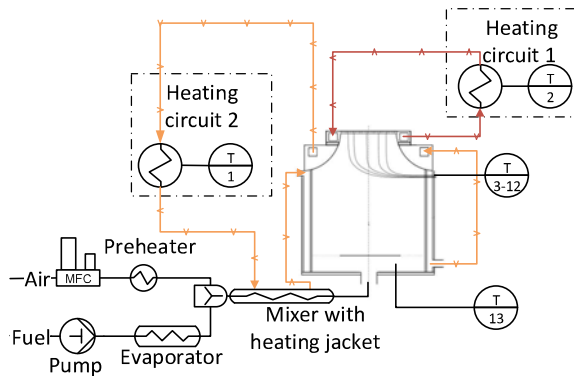


Fig. 2. Setup of the flat flame burner test rig.

The edge of the perforated burner plate is heated with a heating jacket flushed with heat transfer oil to compensate the heat losses of the flame. The corresponding heating circuit 1 (T1) is equipped with an heat circulation thermostat (Peter Huber Kältemaschinenbau AG) which can be adjusted up to a

temperature of 473 K. Upstream of the burner plate, the plenum chamber equipped with a heating jacket serves to distribute the fuel-air mixture. Fuel and air are mixed and tempered further upstream in the supply line. The temperature of the unburned mixture can be adjusted with the heating circuit 2 temperature (T2) and measured via a type-K thermocouple (T13) in the plenum chamber. The liquid fuel is fed via a syringe pump to a direct evaporator (ADROP Feuchtemesstechnik GmbH) and evaporated at 523 K - 553 K. The air flow is controlled by a mass flow controller (Bronkhorst High-Tech B.V.) and preheated with an electrical heater (Leister GmbH) to prevent condensation of liquid fuel components inside the mixer.

Experiments were conducted for four different solvent mixtures, introduced in Table 1 at a preheating temperature of 433 K. The equivalence ratio was varied from 0.6 to 1.3. Maximum values were observed at rich conditions for all formulations ranging from 60 cm/s to 63 cm/s. Flat flames were stabilized at adiabatic conditions for lean mixtures changing to cellular flames with rising equivalence ratio. Fig. 4 presents the measured burning velocities at varying equivalence ratios, distinguishing between cellular and flat flame with circles and squares respectively.

Occurrence of cellular flame structures on flat flame burners were observed also in [15] where the onset of cellularity was explained by hydrodynamic influences within the flame holder [16]. Whereas in [17] the onset of n-butane-air cellular flames was governed by diffusive-thermal mechanisms. Generally cellular flame structures do not comply with the requirements of the Heat-Flux-Method of a flat flame, which can impose uncertainties. The propagation speed of an adiabatic cellular flame corresponds to an average value over a region with varying local temperature and mixture composition with different local burning velocities [18,19].

Definition of Surrogate Fuels

Due to the complexity of the chemical compounds participating in the solvent mixture compositions and the unavailability of appropriate kinetic schemes to account for their combustion behaviour, four customized surrogate solvents are introduced. Each one represents one of the four given solvent formulations.

In order to introduce the customized surrogate solvents, representative species have been selected for each chemical class available in the solvents compositions. The selection of representative species is based on the following criteria: (a) compatibility of molecular structure, (b) simplicity – flexibility and (c) availability of kinetic schemes in the literature.

The portion of the solvent mixtures attributed to aromatics (heavy, light, solvent naphtha, xylene) is represented by mixtures of naphthalene and toluene. N-butanol represents the alcohols in the solvent mixture. Methyl and ethyl acetate account for the esters. The ether content is either attributed to the esters or to the alcohols of the surrogate mixture.

The definition of the solvent surrogates is based on the following criteria: (a) Their compositions must approximate the individual concentrations of each chemical class identified in each solvent's formulation, (b) their heating values must approximate the measured heating value of the specific solvent they represent and (c) their C-H-O content must be similar to the theoretical values of the given solvent formulations.

The proposed compositions for the solvent surrogates are presented in Table 3. Practically, the surrogate solvents are mixtures of the selected representative species (naphthalene toluene, methyl acetate, ethyl acetate, N-butanol), with respect to satisfying the aforementioned criteria. Indicatively, an assumed average 50% of aromatic content of Formulation 1 is attributed to both naphthalene and toluene. The remaining 50% corresponding to the esters, is divided to 20% methyl acetate and 30% ethyl acetate. The individual percentages of naphthalene and toluene as well as the respective individual values of methyl and ethyl acetate as representatives of the esters, are determined via a combined trial and error approach with respect to satisfying the aforementioned criteria. A similar approach has been adopted for all solvent surrogates.

As already mentioned the ether content is either attributed to the esters or to the alcohols of the surrogate mixture. For example, by assuming an initial decomposition step to n-Butanol and ethylene oxide ($C_2H_4O_4$), butyl glycol ($C_6H_{14}O_2$) contained in the solvent formulations 2-4 can be attributed to n-Butanol. However, by assuming a similar decomposition step for other ethers (e.g Dipropylene glycol ether – $C_7H_{16}O_3$) both esters (ethyl acetate) and alcohols (propanol) are produced. In this case, the selection of attributing the ether content to either esters or alcohols was based on satisfying the heating value criterion.

In order to characterize the proposed solvent surrogates' behavior essential mixture properties have been calculated by building simple models in Aspen Plus software. The emerging values are compared with the solvents measured properties and reported in Table 4. Solvent formulation densities were measured using a pycnometer [4]. As it can be seen surrogate mixture densities at ambient temperature conditions are in close agreement with the solvent densities. A slight overprediction within a range of 1.3-3.5% is depicted. The relative content of C, H and O atoms in the surrogate mixtures is also shown since it is an important indicator for the combustion behavior of the examined solvent mixtures as well as for the dimensioning of burners. It can be observed that no significant differences can be observed between C- content of solvent formulations and solvent surrogates. Furthermore, heating values of all surrogates mixtures tend to slightly underpredict the measured solvent values with deviations ranging between 0.8 % to 3.7%.

Table 3. Concentrations (% wt) attributed to the proposed solvent surrogates.

Surrogate Mixture	Aromatics		Esters (Ethers)		Alcohols (Ethers)
	Toluene	aphthalen	Methyl Acetate	Ethyl Acetate	N - Butanol
	[% wt]	[% wt]	[% wt]	[% wt]	[% wt]
Surrogate 1	19	31	20	30	0
Surrogate 2	20	29	5	30	16
Surrogate 3	22	33	12	9	24
Surrogate 4	24	37	6	7	26

Table 4. Density, heating value and C:H:O content: Comparison between measured solvent and predicted solvent surrogate values.

Surrogate Mixture	Density (% from solvent)	Deviation from solvent	C-H-O content	Deviation from solvent	Heating Value	Deviation from solvent
	[kg/m ³]	[%]	[kg/kg _{surr}]	[%]	[MJ/kg]	[%]
Surrogate 1	948.3	1.3	0.73-0.08-0.19	2.8	33.0	-3.2
Surrogate 2	921.0	3.5	0.74-0.09-0.17	1.4	34.8	-3.6
Surrogate 3	918.6	2.1	0.77-0.09-0.14	7.8	36.4	-0.8
Surrogate 4	917.4	2.5	0.79-0.09-0.12	3.8	37.7	-1.6

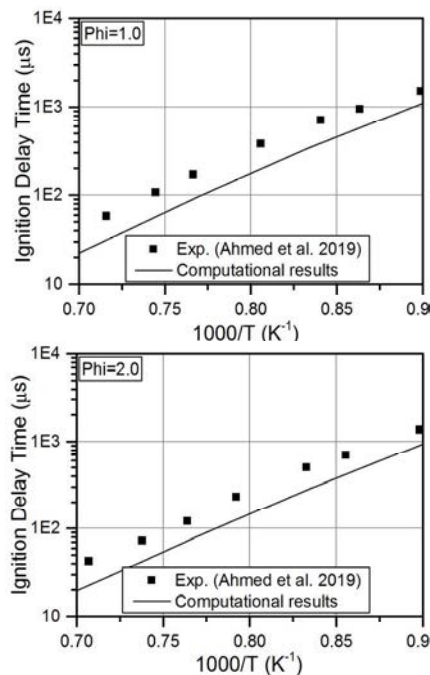
Chemical Kinetics Modelling

A chemical kinetics mechanism has been developed in order to take into account the combustion of solvents. A C₁-C₆ mechanism developed in the Laboratory of Heterogeneous Mixtures and Combustion Systems (NTUA) was utilized as the base chemistry [20,21]. Recent advances showed that the mechanism can very well describe the oxidation of C₂ hydrocarbon fuels, including oxygenates [22] and has also been extended to include models for acetic acid and n-hexane [23].

In this work the mechanism has been expanded with respect to including combustion chemistry for all representative species forming the solvent surrogates. The combustion of naphthalene and toluene selected as representative for aromatics was already included in the base mechanism [23]. Appropriate reaction schemes were incorporated to account for the combustion of methyl acetate, ethyl acetate [24] and n-butanol [25]. Upon selection the respective sub mechanisms were separately incorporated to the base mechanism. Each stage of species oxidation sub mechanism implementation was followed by an individual validation process in order to assess the performance of the expanded mechanism. In its initial form the mechanism includes 258 species and 1342 reactions whereas the emerging mechanism consists of 321 species participating in 1826 reactions.

The methyl and ethyl acetate mechanisms were adopted from the recent work of Ahmed et al. [24] focusing both on the computational and experimental investigation of the respective chemistry and offering appropriate validation targets. Experiments include ignition delay time measurements for three equivalence ratios and at two pressures (p=15-30 bar). The mechanism for methyl acetate adds 10 new species in the base mechanism participating in 23 reactions. The

mechanism for ethyl acetate adds only two species and 46 reactions.

**Fig. 3.** Ignition delay time: Comparison between experimental data [19] and computations with the developed mechanism (p=15 bar).

Computations completed for the referenced study conditions yield a first assessment of the expanded mechanism performance. Indicatively, a comparison with measured ignition delay times at 15bar for two equivalence ratios is shown in Fig. 3 for methyl acetate.

All computations have been performed using the constant pressure reactor module of LOGResearch [26].

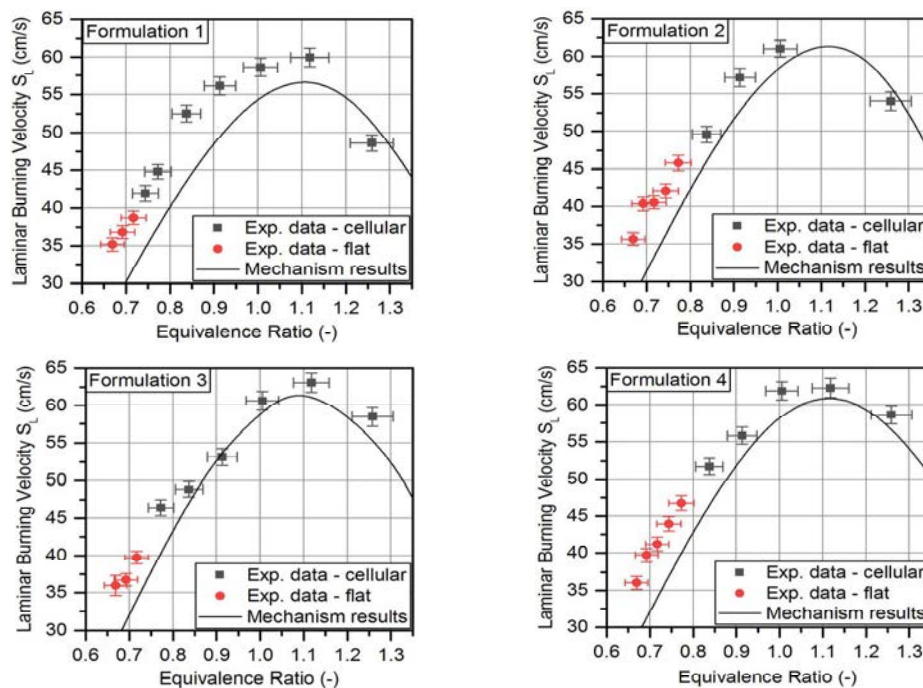


Fig. 4: Laminar burning velocity: comparison between measurements and computational results.

The expanded mechanism gives satisfactory results in the examined cases. A slight underprediction of the predicted ignition delay times is observed.

In order to validate the expanded mechanism ability in accurately reproducing the solvent formulations combustion behaviour, Fig.4 presents a direct comparison between solvent formulations and surrogate mixtures laminar burning velocities. Simulations have been performed with the use of LOGEresearch [26] software in a premixed freely propagating flame for a set of conditions corresponding to the experiments (various equivalence ratios and preheating temperature of 160°C, $p=1\text{atm}$). In all cases the shape of the experimental flame velocity curves is well reproduced by the simulations. Computed maximum flame velocity values are observed, as anticipated, at slightly fuel rich mixtures (approximately $\Phi=1.1$). A very good quantitative agreement between measurements and computations is depicted for at least three solvent mixtures (Formulation 2,3,4) with laminar burning velocity values (measured and computed) reaching maximum at slightly above 60 m/s. In the case of Formulation 1 quantitative discrepancies are observed and simulations tend to underpredict measurements by 10-15%. Overall, a satisfactory agreement is established between experimental values and laminar burning velocity predictions obtained by the developed mechanism.

Conclusions

This work presents a combined experimental and chemical kinetics modelling approach to account for the combustion behaviour of solvents utilized in coil coating processes. Solvent vapours are complex mixtures of

organic compounds and can be potentially used as fuel for process heat production.

Initially, heating values and laminar burning velocities of typical industrial solvent formulations comprising alcohols, ethers, esters and aromatics are experimentally investigated. Measurements with the use of an oxygen bomb calorimeter show that the examined formulations have higher heating values in the range between 34-38 MJ/kg. Furthermore, laminar burning velocities of solvent mixtures at a preheating temperature of 160°C and at atmospheric pressure for equivalence ratios of $\phi=0.65-1.25$ were determined by the Heat-Flux-Method using a flat flame burner. Maximum laminar burning velocities in the range of 60-65 cm/s are obtained for all formulations at equivalence ratios around $\phi = 1.1$.

Due to the complexity of species participating in the solvent formulations and the unavailability of kinetic schemes to describe their oxidation, typical surrogate solvents are introduced, one for each formulation.

An “in-house” chemical kinetics mechanism has been extended in order to take into account the solvents’ combustion. Appropriate reaction schemes were incorporated to account for the combustion of methyl acetate, ethyl acetate and n-butanol. Each stage of species oxidation sub-mechanism implementation was followed by an individual validation campaign in order to assess the mechanism expansion process. The emerging mechanism consists of 321 species participating in 1826 reactions and its overall performance is validated against the aforementioned laminar burning velocity measurements. A good qualitative and quantitative reproduction of the experimental curves is depicted with maximum discrepancies observed in the range of 10-15%.

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