Empirical Model to Predict Freezing Time for different range of DEF Tank Volumes used in SCR Systems

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ABSTRACT

Introduction of stringent emission norms has lowered the allowable limits for Oxides of Nitrogen (NOx), coming out of Diesel engine exhaust. This has led to advances in the after-treatment system with development of reduction strategies for NOx. Selective Catalytic Reduction (SCR) is one such system. The automotive industry has accepted use of an aqueous urea solution popularly known as Diesel Exhaust Fluid (DEF or Adblue©) as a reductant for this purpose. DEF containing 32.5 % urea & 67.5% deionized water forms a eutectic mixture thereby pushing the freezing point of DEF to 12°F (or -11°C). Use of an insulating blanket around the tank and/or placement of tank near engine might delay the freezing of DEF. However when subjected to sub-freezing temperatures for a longer duration DEF freezes entirely. If DEF remains in frozen state, engine out emissions tend to exceed the regulatory limits.

Environmental Protection Agency (EPA) has set guidelines for carrying out a standard thawing procedure in order to determine whether a particular engine satisfies the emission norms or not. EPA test procedure requires the DEF tank to be soaked for a period of 72 hours at -17.78°C before the thawing test is carried out. However the time taken to freeze DEF varies with the tank volume and dimensions and also on the tank material. Small tanks might freeze before this time period while larger tanks might contain some amount liquid DEF even at the end of 72 hours. Incomplete freezing of DEF at the start of thawing test leads to over-prediction of melt fraction of DEF. Also when melting phenomenon is modelled using Computational Fluid Dynamics (CFD) tools, it is assumed that liquid is completely in frozen state at the start of melting. This leads to error in prediction of melt volume since there is possibility of residual molten pocket. Hence determination of freezing time is important. Here an empirical model has been identified from literature for determining time taken for freezing the complete volume of DEF. The freezing time has been correlated with the solidified fraction of DEF using dimensionless factors commonly associated with heat transfer during freezing phenomenon, that is, Fourier, Stefan and Rayleigh numbers. The model is aimed at predicting the freezing time in common geometries of rectangular and cylindrical enclosure. This model has been validated with simulation as well as experimental results known in the literature. CFD simulation modeling strategy of transient freezing and its comparison with test is also compared in this study.

KEYWORDS

Freezing phenomenon, Diesel Exhaust Fluid, Empirical Model.

NOMENCLATURE

NO_x	Oxides of Nitrogen
SCR	Selective Catalytic Reduction
DEF	Diesel Exhaust Fluid
EPA	Environmental Protection Agency
DOC	Diesel Oxidation Catalyst
DPF	Diesel Particulate Filter
AT	After-treatment
AUS	Aqueous Urea Solution
EGR	Exhaust Gas Recirculation
CFD	Computational Fluid Dynamics
Ra_{cr}	Critical Rayleigh number

Ra	Rayleigh number, $Ra = \frac{g\beta(T_h\text{-}T_m)L^3}{\alpha\nu} Ra = \frac{g\beta(T_h - T_m)L^3}{\alpha v}$
Fo	Fourier number, $\boldsymbol{Fo = \frac{\alpha t}{L^2}}$
A	Aspect ratio, ratio of length to width
Ste	$Ste = \frac{C_p(T_h - T_m)}{H}$ Stefan number,
τ	Dimensionless time, Ste*Fo
g	Acceleration due to gravity, 9.81m/s^2
T	Temperature
L	Characteristic length (length in case of a rectangular enclosure or radius in case of a cylinder)
t	Time
C_p	Specific heat at constant pressure, kJ/kg-K
H	Enthalpy of fusion, kJ/kg
α	Thermal diffusivity
v	Kinematic viscosity, m^2/s
ρ	Density, kg/m^3
β	Volumetric expansion coefficient, 1/K

INTRODUCTION

The introduction of norms for governing the pollutant limits from engine exhaust has led to development of after treatment system of diesel engine over the years. Figure 1 shows a schematic progression of diesel after-treatment (AT) over the years. It can be seen that Selective Catalytic Reduction has become a critical part of the AT system as per the recent emission norms since 2010.

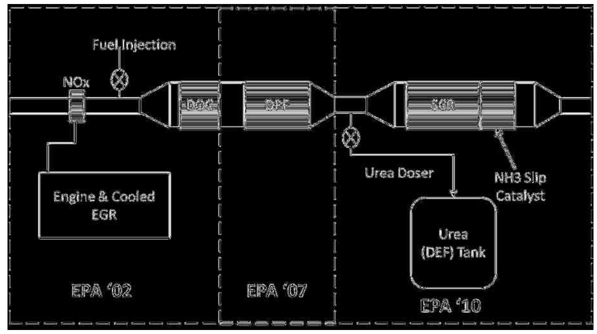


Fig 1- Evolution of after-treatment system as per EPA norms

Since its induction in the AT system, SCR technology has undergone a lot of technological advances to adapt it into mobile engine applications, thereby making it a robust and easy to use system. Figure 2 shows the emission regulations for NO_x and PM over the years [1]. It can be seen that the allowable limits for oxides of Nitrogen (NO_x) and particulate matter (PM) have been drastically reduced. Out of these two, elimination of NO_x is critical and hence stricter norms have been imposed over it. An efficient SCR system having high conversion efficiency can successfully reduce NO_X over 90% [2]. Reduction of NO_X is achieved by using Ammonia as a reductant. For onboard applications, Ammonia is stored in the form of aqueous urea solution (AUS), commonly known as Adblue© or Diesel Exhaust Fluid (DEF). DEF constitutes liquid urea (32.5%) and water (67.5%) [3].

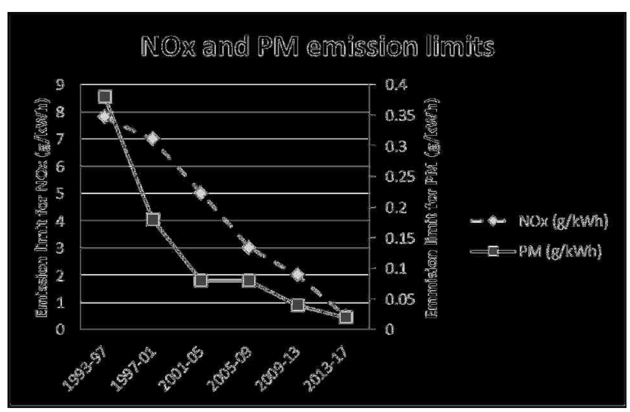


Fig 2- Emission limits for NOx and PM over the years

DEF is introduced in the exhaust stream, as shown in figure 3, injected as a pressurized liquid. This high pressure combined with elevated temperature from the exhaust gases lead to thermal breakdown of DEF, thereby generating Ammonia, which reduces NO_x to Nitrogen (N_2) [4].

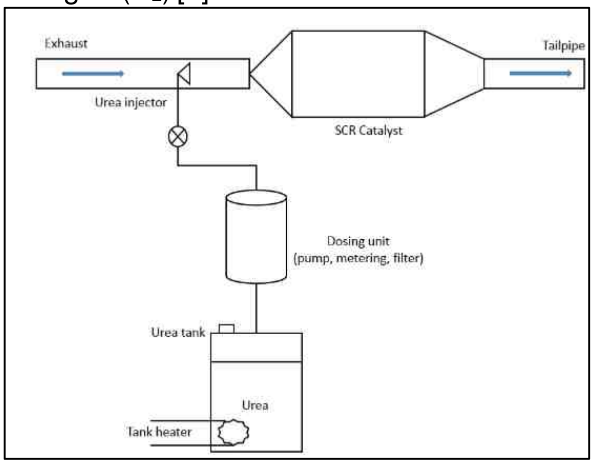


Fig 3- Schematic diagram of an SCR system

Presence of DEF poses a major concern at cold temperatures, since its freezing point is -11℃ (or 12℉). Thus when ambient temperature reaches sub-freezing temperature there is a hindrance in operation of SCR. To avoid blockage of pipes due to frozen DEF, tanks come with a provision of purge [5]. This ensures that when the vehicle stops, DEF present in the transport lines is sent back to the tank. This ensures that no damage is done to the dosing and supply units carrying DEF. As per US EPA regulations, sufficient quantity of DEF has to be thawed within 70 minutes of vehicle start. EPA has also set guidelines to carry out a thawing test for validating DEF tank design and thawing performance criteria.

Two types of test procedures are provided- under the first test setup the entire engine system is put into a cold cell and run at specific conditions and under second type pass/fail criteria for tank-only validation is given. In the second type of setup only the DEF tank is put into the cold cell and external pumps and heaters are used to simulate engine running conditions [5]. The tank is cold soaked at a temperature of at least -17.7 ℃ (0 ℉) for 72 hours until tank reaches a solid DEF state. It is assumed at the end of soaking period that there are no liquid pockets remaining. However in practical scenario due to non-uniform cooling and inaccuracy of temperature measurement, there is a possibility that DEF is not frozen completely. Also for tanks with smaller capacity freezing might occur a lot earlier causing wastage of refrigeration energy. Using Computational Fluid Dynamics (CFD) tools for studying the liquid–solid interface as well as freeze volume during freezing is time-consuming and there is a huge amount of uncertainty because of unpredictable nature of natural convection. The objective of this study is to predict the time taken for the entire volume of DEF to be frozen and reduce this uncertainty in prediction of freezing time. This is accomplished by using empirical models developed in the literature for the phenomenon of uniform cooling. Here a simple rectangular geometry is considered for prediction of the freezing time. To ensure the validity of empirical model, it has been compared with some literature as well as experimental data to understand its applicability. Transient freeze CFD simulation strategy and comparison with test has been discussed in the last section of this paper.

LITERATURE REVIEW

Freezing phenomenon

In order to identify and apply a suitable empirical model for prediction of freezing time it is important to understand the physical phenomenon of freezing and the factors governing this process. The factors which govern any heat transfer phenomenon are the geometry in consideration (characteristic length and/or aspect ratio), the temperature difference, the time for which the freezing is observed & the thermophysical properties of the melting fluid [6]. Also identifying the dominant mode of heat transfer for a particular set of problem is necessary. It is observed that as freezing progresses, natural convection occurs in the unfrozen liquid into which the solidification front advances, until the temperature of the liquid exceeds the phase-change temperature [7]. A detailed examination using flow visualization experiments led to better understanding of buoyancy-induced fluid motion and its effect on the shape and motion of the solid-liquid interface [8]. Here the authors observed the simultaneous motion of cooled fluid parcels and heated fluid parcels. The cooled fluid parcels moved from the cooled top plate toward each of the vertical side walls while the heated fluid parcels rising from the heated bottom plate and moved toward the central region of the cell. Over a period of time, it was observed that pair of well-established, two-dimensional convection rolls was formed. These rolls were symmetric and persisted during the entire process of freezing. Also it was observed that presence of such convection rolls was same for various aspect ratios during freezing, which the authors attributed to the stabilizing effect of the freezing front solid. Heat conduction in the solid plays a dominant role in controlling the motion and shape of interface during the initial period of the freezing process because of the large temperature gradient in the solid.

M. Akyurt et. al reviewed the characteristics of solidification and melting for water [9]. The authors have identified four distinct stages of freezing as super-cooling, nucleation and the formation of dendritic ice, the growth of concentric rings of solid ice at 0℃ and the final cooling of the solid ice. In general, it can be seen that dimensionless numbers play an important role when an empirical model needs to be developed. In studies where free or natural convection is considered, a linear relation is established between melt volume and dimensionless numbers like Rayleigh, Stefan and Fourier numbers and the correlation is typically of the type: [10-13]

$$\frac{V}{V_o} = C_1 Ra^a C_2 Fo^b C_3 Ste^C \qquad \text{(equation 1)}$$

Where V: Volume of frozen fluid melted after some time, t

V_o: Initial volume of the frozen liquid

C_1, C_2, C_3 & a, b, c: Coefficients whose value vary depending upon geometries

The significance of Ra, Ste & Fo numbers has been explained in the following section.

Significance of dimensionless numbers-

(i) Rayleigh Number (Ra):

Rayleigh number plays an important role in case of freezing because of the liquid state of the fluid in consideration. The predominant heat transfer phenomenon as the freezing progresses has been identified as natural convection as understood from previous section. The advent of convection currents inside the fluid depends on (a) Temperature difference the fluid is subjected to; (b) Thermophysical properties of the fluid; (c) Geometric factors for the system in consideration. For a DEF tank soaked in the cold chamber, providing a lower temperature than the freezing point ensures quick advent of solidification. In case of cold soaking of DEF tanks, the fluid (that is DEF) near the boundary of the tank starts to cool first and slowly cooling progresses towards the center of the geometry. This temperature difference between the fluid in the center and outer boundaries sets the convection current in motion. The ease with which these currents travel across the fluid depends on its viscosity in case of pure natural convection.

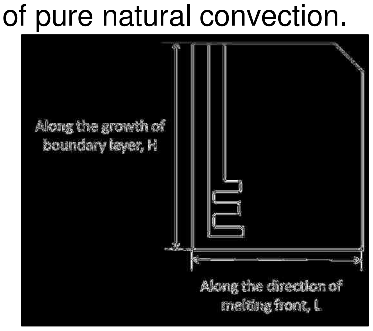


Fig 4- Significance of characteristic length in calculation of dimensionless numbers

In doing so the fluid also has to overcome viscous or drag forces. These viscous forces arise due to the internal resistance of fluid layers to oppose motion. Thus in order to enable the energy transfer between the molecules of top and bottom layer, the fluid has to overcome viscous forces. Thus convection, where Ra >> 1, signifies the phase in heat transfer during which buoyant forces dominate the viscous or drag forces. Mathematically, it is expressed as-

$$Ra = \frac{g\beta\Delta T H^3}{\alpha\nu} \qquad \text{(equation 2)}$$

The height, H, signifies the progression of boundary layer as shown in figure 4.

(ii) Fourier Number (Fo):

The dimensionless number which factors in time is Fourier number (Fo). Mathematically, it is expressed as-

$$Fo = \frac{\alpha t}{L^2} \qquad \text{(equation 3)}$$

Thermal diffusivity, α (=k/ρC_p), is a ratio of heat conducted to the heat stored. Hence a higher the value of thermal diffusivity indicates faster heat propagation and thus characteristic length in case of Fo number is taken as the length of tank along which the solid-liquid interface advances as seen in figure 4. In most studies, Fo number is also referred as dimensionless time.

(iii) Stefan Number (Ste):

Stefan number (Ste) depends predominantly on the thermophysical properties of the material and represents a phase change phenomenon of either melting or freezing. It is defined as-

$$Ste = \frac{C_p \Delta T}{H} \qquad \text{(equation 4)}$$

Here, in equation (4), the numerator ($C_p \Delta T$) signifies the energy given by sensible heat while the denominator (H) signifies the latent heat energy. Hence Ste number signifies the contribution from each of the enthalpies during the process of melting. A higher value of Stefan number indicates that phase change phenomenon is predominantly governed by the change in sensible energy, while a lower value signifies more energy is required for the phase change, since melting enthalpy is higher.

CORRELATION STUDY

The DEF tank sizes depend on the range of application. Tanks can be classified on the basis of (a) Quantity (b) Orientation (portrait: when height is more than length or landscape: when height is less than length) and (c) heating arrangement (either engine coolant or electric heating element). The size of DEF tanks vary from a portable 5 gallons disposable tank to a 330 gallons refillable tote. However this study is focused on DEF tank used for onboard application typically for mid-range (64-300 kW or 85-400 hp), heavy- duty (401-750 hp or 300-560 kW) and a few High Horsepower (>560 kW or 751hp) engines as well. While the mid-range engines employ smaller DEF tanks having a capacity of 5 to 30 gallons (19-114 litres), heavy-duty & HHP engines can have a DEF tank volume of up to 100 gallons (375litres). Table 1 provides the range of dimensionless numbers considered in this study.

Table 1- Range of parameters

Stefan number	0.56-1.32
Fourier number	0.44-9.29
Rayleigh number	3.44e^10 – 2.08e^12
Nusselt number	42

This study is focused on calculation of freezing time in a rectangular enclosure for simplest geometry of DEF tanks. Though most of the research work has been carried out using water/ice as the fluid, its application to DEF is justified if the values of dimensionless numbers fall within the range. Many studies have been conducted to correlate freeze volume as a function of dimensionless numbers Ra, Ste, Fo and/or A [10-13]. The earliest of such studies was conducted by W. Z. Cao et. al [10]. They conducted an experimental study on the transient solidification of water in a rectangular inclined cavity with one cold wall and five adiabatic walls. The dominance of natural convection during solidification phenomenon was indicated by the temperature distribution in the cavity and the growth of the ice/water interface. The following correlation was developed in this study:

$$\frac{V}{V_o} = 3.515\, \tau^{0.671} e^{-0.066\cos(\theta - 41.92)}$$

(equation 5)

However in practical scenario, cooling of the DEF tank occurs uniformly over all walls of the tank and hence application of this model tends to over-predict the freezing time since it has considered only three out of four walls to be adiabatic. Also the authors here have neglected the role of natural convection which is indicated by the absence of Ra number term in equation (1).

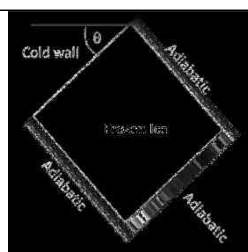
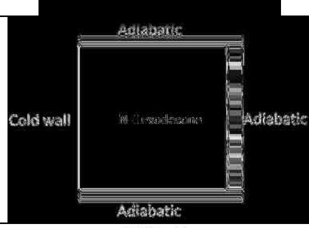
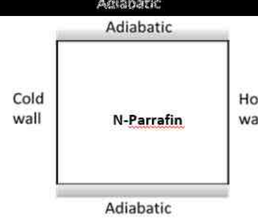
Furthermore, under similar experimental conditions, the study of effects of initial liquid superheat and aspect ratio on the freezing characteristics has been carried out previously [13] by observing freezing of PCM (n-hexadecane) in a rectangular enclosure has been investigated. Following empirical correlation was developed:

$$\frac{V}{V_o} = 1.897\ \tau^{0.556} Ra^{0.004} A^{0.271} \qquad \text{(equation 6)}$$

The authors observed a slight decrease in the solidified mass fraction with an increase of initial liquid superheat, indicated by a small value for Ra number. Also a positive value for aspect ratio indicated larger solidified mass fractions were observed for higher aspect ratios. Similar observations were made in the past [11]. A numerical study was conducted and solidification process was found to be affected by the natural convection at the early stage of the solidification than at the later stage. The aspect ratio of the enclosure had limited effect on the shape of the shape of interface and a negligible effect on the freezing volume.

In order to further study the role of natural convection during solidification, study on solidification of a pure metal [12] was done. The experimental test cell was rectangular having two opposing sidewalls held at constant but different temperatures, while the remaining walls are well insulated. A numerical model was also used for observing the freeze fraction and to compare its results with the experimental data. The importance of natural convection in the liquid metal on the temperature distribution in the melt as well as on the shape and motion of the solid-liquid interface is inferred from

Table 2- List of important correlations and paper

AUTHOR/YEAR	BOUNDARY CONDITIONS	CORRELATION DEVELOPED	% Error using Cao et al	% Error using Leong et al	%Error using Wolff et al
W. Z. Cao and D. Poulikakos, 1991 [10]		$\frac{V}{V_o} = 3.51\ \tau^{0.671} e^{-0.066\cos(\theta-41.92)}$	-	-40.56	-25.46
K.C. Leong and F.L. Tan, 1997 [13]		$\frac{V}{V_o} = 1.897\ \tau^{0.556} Ra^{0.004} A^{0.271}$	31.12	-	29.13
F. Wolff And R. Viskanta, 1998 [12]		$\frac{V}{V_o} = 2.91\ \tau^{0.53} Ra^{-0.05} A^{-0.36}$	15.12	17.21	-

measurement of temperature and solid-liquid interface position. The variation of the solidified volume fraction with time has been determined by integrating the interface contour. Non-linear regression was used to obtain a best curve fit represented by equation (7) by using and employing dimensionless time (Ʈ), Ra number and aspect ratio as the dimensionless parameters.

$$\frac{V}{V_o} = 2.91\ \tau^{0.53} Ra^{-0.05} A^{-0.36} \qquad \text{(equation 7)}$$

The experimental data was found to collapse quite onto a single correlation given in equation above. The exponent of the dimensionless time of 0.53 is close to the exponent found for pure conduction solution of 0.5 in case of melting. A negative exponent was obtained for aspect, indicating that the solidified volume fraction decreases with increasing Rayleigh numbers and aspect ratios.

The empirical models discussed here (equation 5-7) have been used for prediction of freeze volume. These models have been utilized for prediction of freezing time assuming that the freeze fraction is 1, that is $V/V_o = 1$. The application of these models has been done with following set of assumptions:

(i) The concentration of DEF over the freezing process is considered uniform. Also the thermophysical properties are assumed to be independent of temperature.
(ii) DEF tank is considered as 2D model considered; the progression of solidification is considered to be a one-dimensional phenomenon.
(iii) It is assumed that DEF tank is cooled uniformly from all four sides and thus freezing takes place uniformly, that is, at isothermal condition.
(vi)The thermophysical properties used are enlisted in table 3.

These values are widely accepted and used in most of the studies conducted on DEF [14].

Table 3 -Properties of DEF

Chemical formula	$(NH)_2CO$
Melting temperature (°C)	-11
Density, solid (kg/m^3)	1010
Density, liquid (kg/m^3)	1090
Specific latent heat (kJ/kg)	152.86
Specific heat, liquid (kJ/kg-K)	1.6
Specific heat, solid (kJ/kg-K)	3.4
Thermal conductivity, solid (W/mK)	0.75
Thermal conductivity, liquid (W/mK)	0.57

RESULTS

In order to understand applicability of a correlation and selecting the robust one is important. Results are compared with experimental results from similar literatures. This has been summarized in table 2. It can be seen that out of the three enlisted correlations, [12] gives better results when applied to the two other studies. It can be accomplished that this empirical relation is valid for water as a fluid.
To validate its application in case of DEF tanks it is important to apply this empirical model and compare with experimental results of DEF tanks of different sizes.

DEF tank volume	Freezing time (hr)	Cao et al correlation	% Error	Wolff et al correlation	% Error	Leong et al correlation	% Error
9	90	8.19	90.9%	29.17	67.6%	6.25	93.1%
18	146	16.25	88.9%	175	19.9%	81.67	44.1%
25	237	22.5	90.5%	243.6	2.8%	122.92	48.1%

Fig 5- Comparison of different approaches for validation of DEF tank results

This particular model (eq. 7) has been applied to DEF tank results as published in the study by Stefan aus der Wiesche [14]. In the literature CFD simulation was carried out on different tank geometries and those results were compared with experiment and it showed prediction of freeze time within 20% for larger tank volumes which is reasonable looking at lot of other phenomena happening in 3D. Hence, empirical model as mentioned in eq. 7 was used to compare the results between experiment and CFD for DEF. Since, earlier comparison was done for water but this empirical model will be used for DEF tanks, so this comparison is very important to understand if it can be used for SCR application. Experiment as mentioned in [14] was done on three different tank volume ranging from 9, 18 and 25 liters. The percentage error in case of freezing time for three tank volumes that is

9, 18 and 25 liters have been compared here. The error percent in case of 25 liters is less which signifies the dominant role of Ra number as expressed in the correlation. However it can be seen that for smaller tank volume the error percent is very high. This is because conduction plays an important role for smaller tanks and heat transfer is dominated by conduction. As stated before for SCR application typical tank sizes would start from 19 liters and then it would go up to 335 liters for HHP application hence, this correlation would be useful in designing DEF tanks and executing freeze tests for those application.

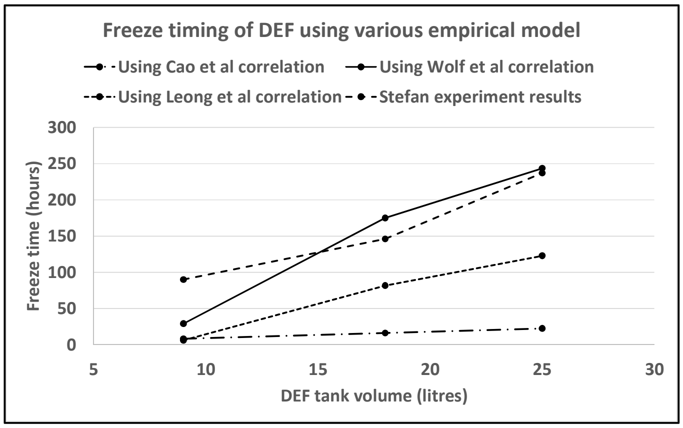


Fig 6- Comparison of various approaches

CFD SIMULATION RESULTS

Till now in this paper most of the work discussed has been associated with identifying empirical model which is valid for SCR application and understanding its validity by comparing the results with different experiments for water and DEF. As mentioned before empirical model assists in predicting the freeze time but sometimes it is important to visualize the freezing sequence in the closed cylinder. So, comparison of CFD simulation with test was also done to understand how much close we can predict the temperature variation. The objective here was to validate our simulation processes. Hence, a simple cylindrical cavity was designed especially to conduct test at Cummins Doser Lab located at Germany and thermocouple dipped inside the volume along the center axis to understand the freezing behavior wrt time. Specimen was cooled inside a climatic chamber and data of thermocouple was logged. Challenge here is to correlate the transient behavior of DEF freezing. DEF cylindrical cavity as shown in Fig 7 is considered for this experiment. The dimensions are considered as per it's validity mostly for DEF dosing systems.

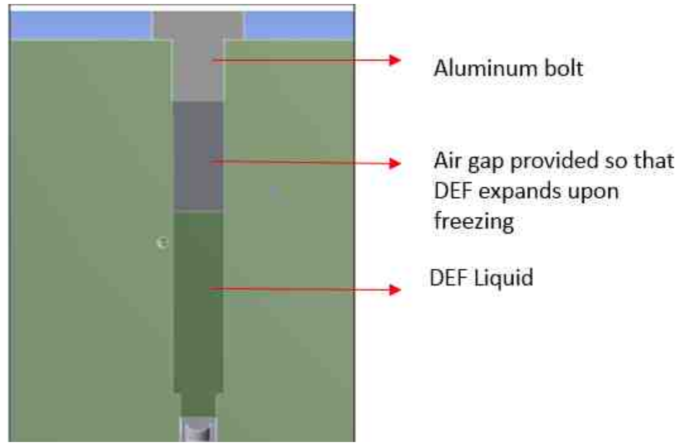


Fig 7 Simplified model considered for validation with test

Modeling in CFD requires meshing the geometry and then using case setup where in DEF properties were used from BASF leaflet [3]. Below mentioned are the key modeling assumptions considered for conducting this simulation.

- k-epsilon turbulence model, since there is no significant turbulence regime developed in this type of problem but still this model has been used. However, laminar model would also be enough to execute such analysis and results can be quick to compute.
- Solidification-Melting model switched on FLUENT. This model assists in calculating the liquid fraction which is a function of latent enthalpy. Hence, as the temperature changes the liquid fraction updates for each node. FLUENT uses below equation to compute liquid fraction where L is latent heat and β is liquid fraction which is computed based on temperature achieved during simulation at each node.

  $\Delta H = \beta L$ equation 8
- All solid and liquid volumes provided with relevant material properties
- DEF properties considered from BASF leaflet with time variation for specific heat attached to it
- Density and thermal conductivity considered constant values
- Standard initialization

- All outer walls attached with transient table from experiment data

Outer walls have been attached with the transient table from test which is varying from 30 ℃ to -40 °C. Figure 8 shows that the correlation between CFD simulation and test is very good. The trend as well as absolute values shows acceptable match. The time average difference between CFD and test is 2.36 ℃ with maximum going up till 8.22 ℃. We can also observe that freezing is moving in wards from walls to the center of the core as shown in Figure 9.

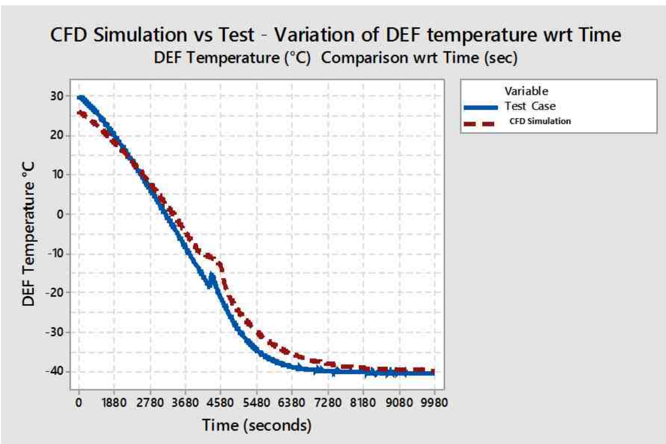


Figure 8 CFD Simulation results comparison with Test data

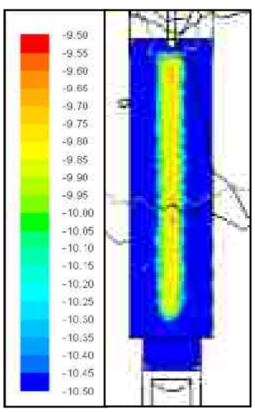


Figure 9 Static Temperature Contour Plot showing DEF freeze behavior in cylindrical tube

We also observed that at -18 ℃ there is slight peak in temperature in DEF volume this is due to phase change from liquid to solid. This type of behavior has been observed in many literature which talks about freezing behavior in water. This peak is visible in CFD results as well but does not match the exact time as observed in test. This is dependent on many factors and is always difficult to correlate. But the phenomena of phase change is well captured in simulation.

CONCLUSIONS

The main aim of this paper is to identify correlation for prediction of freeze time which can be used while conducting freeze test for DEF tank. CFD simulation takes time hence there is need for a correlation which can provide results quickly within acceptable error band for typical SCR applications. Various literature were studied to identify correlation which are valid for Mid-range and HHP application DEF tanks. It was observed among various empirical relation studies, the correlation defined by [12] is the most robust one. On comparing results with other experimental data provided in literature it could give results within 20% error band which is reasonable. But since most of the literature used fluid different than Def hence, it was important to correlate results with experiment done using DEF. [14] has conducted such study in the past where data for freezing was generated using different tank volumes was done. Correlation from [12] was used and results were within acceptable limit of less than 25% and correlation improved as tank volume increased.

If the accuracy is required to be improved in prediction and if the volumes are very small then CFD simulation can be used to predict freeze time and it also helps to visualize the phase change behavior. Hence, comparison of CFD simulation with test was done which showed correlation within 2% and average temperature deviation of 2.36 ℃ between simulation and test wrt time. Simulation also matched the trend and phenomena of freezing accurately. So, depending on the requirement of accuracy and tank volumes any method can be chosen to predict freezing time and behavior.

FUTURE STUDY

This empirical model was validate up to 25 lit DEF tank size as discussed in above section. It has been observed that as tank sizes increases correlation with test improves. So, in future more work is required to understand the robustness of empirical model for larger tank sizes probably up to 375 liters.

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