6 Conclusion & Scope for Future Work

6.1 CONCLUSIONS

In the present work, using ternary phase diagrams, the submerged arc welding fluxes were designed for pipeline steel. Fluxes were formulated for basic, rutile-basic, and rutile-acidic flux systems. Mathematical regression modeling of different physicochemical and thermophysical properties performed using extreme vertices design approach. Multi-pass bead on plate weld deposit experimentation was performed using basic, rutile-basic, and the rutile-acidic fluxes. Mathematical regression modeling of bead chemistry, average grain size, and microhardness for three flux systems evaluated. A comparative study of chosen fluxes (selected from three flux systems) with that of commercial available submerged arc welding flux was carried out.

6.1.1 Conclusions from study on development of SAW fluxes

- Regression analysis indicates that individual flux components show antisynegistic effect on grain fineness number for basic, rutile-basic and rutile-acidic flux systems. Binary components such as CaO.SiO₂, SiO₂.CaF₂ and SiO₂.Al₂O₃ show synergistic effect and thus increase the grain fineness number while CaO.CaF₂, CaO.Al₂O₃ and CaF₂.Al₂O₃ show antisynergistic effect and thus decrease it for basic flux system. Other binary mixture components TiO₂.CaO, SiO₂.CaO, SiO₂.Al₂O₃, TiO₂.MgO, TiO₂.Al₂O₃, SiO₂.Al₂O₃ and MgO.Al₂O₃ decrease the grain fineness number for rutile-basic and rutile-acidic flux systems. Ternary mixture components SiO₂.CaO.Al₂O₃, TiO₂.SiO₂.Al₂O₃ and SiO₂.MgO.Al₂O₃ show synergistic effect on grain fineness number while TiO₂.MgO.Al₂O₃ and TiO₂.Al₂O₃. (TiO₂-Al₂O₃) show antisynergistic effect on it in rutile-basic and rutileacidic flux systems.
- Individual flux components decrease the density for basic and rutile-basic flux systems, while for the rutile-acidic flux system, they increase it. Binary component SiO₂.Al₂O₃ increases the density for basic and rutile-acidic flux systems. Binary mixture components CaO.CaF₂, SiO₂.CaF₂, CaF₂.Al₂O₃, TiO₂.CaO and MgO.Al₂O₃ increase the density and show synergistic effect while CaO.SiO₂, CaO.Al₂O₃, TiO₂.SiO₂, TiO₂.MgO, TiO₂.Al₂O₃ and SiO₂.MgO decrease it and show antisynergistic effect for basic and rutile-acidic flux systems. Ternary mixture components CaO.CaF₂.Al₂O₃ and SiO₂.MgO.Al₂O₃ increase the densitv and show synergistic effect while CaO.SiO₂.CaF₂, CaO.SiO₂.Al₂O₃, SiO₂.CaF₂.Al₂O₃, TiO₂.SiO₂.MgO, TiO₂.SiO₂.Al₂O₃ and TiO₂.MgO.Al₂O₃ decrease it for basic and rutile-acidic flux systems.
- Regression analysis shows that individual flux components increase the weight loss for a basic flux system, while for rutile-basic and the rutile-acidic flux systems they decrease it. SiO₂.Al₂O₃ is the binary mixture component, which increases the weight loss in basic as well as in the rutile-basic flux system, while for the rutile-acidic system, it decreases the weight loss. CaO.SiO₂ binary mixture component increases the weight loss in the basic flux system while for rutile-basic flux system it decreased. Binary mixture component CaO.Al₂O₃ shows a negative effect on weight loss in basic flux system, while for the rutile-basic flux system, while for the rutile-basic flux system, it shows a positive effect. Binary mixture TiO₂.Al₂O₃ and TiO₂.MgO show synergistic effect on weight loss for rutile-basic flux systems. CaO.CaF₂, SiO₂.CaF₂, CaF₂.Al₂O₃, TiO₂.SiO₂, TiO₂.CaO, SiO₂.MgO and MgO.Al₂O₃ binary mixture components show a negative effect on it for basic, rutile-basic

and rutile-acidic flux systems. Ternary mixture component $TiO_2.SiO_2.Al_2O_3$ and $TiO_2.CaO.Al_2O_3$ increases the weight loss for rutile-basic flux system while ternary component $SiO_2.MgO.Al_2O_3$ decreases it for rutile-acidic flux system.

- Individual flux components show a synergistic effect on change in enthalpy for basic and rutile-basic flux systems, while for the rutile-acidic flux system they decrease it. SiO₂.CaF₂ is the binary component which increases the enthalpy for basic flux system. TiO₂.SiO₂ and TiO₂.Al₂O₃ increases the enthalpy for the rutile-basic flux system while it decreased for rutile-acidic flux system. TiO₂.CaO shows a positive effect while TiO₂.MgO, SiO₂.MgO and MgO.Al₂O₃ show a negative effect to change in enthalpy for rutile-basic and rutile-acidic flux system. SiO₂.Al₂O₃ is the only binary component which decreases the enthalpy in all the three flux systems while binary mixture component SiO₂.CaO and CaO.Al₂O₃ decrease it in basic and rutile-basic flux systems. TiO₂.SiO₂.Al₂O₃ is the ternary mixture component, which increases the enthalpy for rutile-basic and rutile-acidic flux systems.
- Individual flux components increase the thermal conductivity for the basic flux system, while for rutile-basic and rutile-acidic flux systems individual flux components decrease the thermal conductivity. Binary mixture component SiO₂.Al₂O₃ shows a synergistic effect while CaO.SiO₂ shows antisynergistic effect on it for basic and rutile-basic flux system. TiO₂.Al₂O₃ binary component increases the thermal conductivity in rutile-acidic flux system while for rutile-basic flux system it decreased. Binary mixture components CaO.CaF₂, CaO.Al₂O₃, SiO₂.CaF₂ and TiO₂.CaO decrease the thermal conductivity for basic and the rutile-basic flux systems. All the ternary mixture components decrease the thermal diffusivity is increased by individual flux components while for the rutile-basic and rutile-basic flux system. In basic flux system thermal diffusivity is increased by individual flux components while for the rutile-basic and rutile-basic flux systems, it shows an antisynergistic effect.
- Binary mixture components SiO₂.Al₂O₃ and TiO₂.Al₂O₃ increase the thermal diffusivity both in rutile-basic and rutile-acidic flux systems, while CaO.SiO₂ decreases the thermal diffusivity in the basic system but for rutile-basic flux system it increased. Binary components CaO.Al₂O₃, TiO₂.SiO₂ and MgO.Al₂O₃ show a positive effect on thermal diffusivity for rutile-basic and the rutile-acidic flux systems. In all the three flux systems binary components CaO.CaF₂, SiO₂.CaF₂, TiO₂.CaO and SiO₂.MgO show antisynergistic effect on thermal diffusivity. The ternary mixture component increases the thermal diffusivity for rutile-basic and the rutile-acidic flux systems, while basic flux system is not affected by the ternary mixture components.
- All the individual flux components decrease the specific heat for basic, rutile-basic and the rutile-acidic flux systems. Binary mixture component CaO.SiO₂ increases specific heat for basic system while for rutile-basic flux system it decreases the specific heat. Binary component SiO₂.Al₂O₃ and TiO₂.Al₂O₃ decreases specific heat for rutile-basic system while for rutile-acidic flux system it shows positive affect. TiO₂.SiO₂ binary component increases the specific heat for both rutile-basic and rutile-acidic flux systems while remaining binary mixture components decrease the specific heat for rutile-basic flux system. CaO.CaF₂ and MgO.Al₂O₃ increase the specific heat for basic and rutile-acidic flux systems. TiO₂.SiO₂.CaO is the only ternary mixture component which decreases the specific heat while all the remaining ternary mixture components increase the specific heat for basic, rutile-basic and rutile-acidic flux systems.

6.1.2 Conclusions from Wettabillity study of SAW fluxes

• Measured contact angle value increased with increasing $TiO_2/MgO \& TiO_2/Al_2O_3$ flux ratio. Flux numbers 3, 9, 10, 16, 17, & 18 give an optimum value of measured contact angle due to lesser spreading area over the heating substrate. Flux 7 gives a very lower value of contact angle ($\theta = 0^\circ$) and due to higher value of the spreading area. Lower contact angle provides the higher wettability between the flux and the heating substrate.

- With an increase of the TiO₂/SiO₂ ratio up to 1.5 to 2.0, the calculated surface tension value is decreasing while, after that, it increased with an increase in the TiO₂/SiO₂ ratio. It means that surface tension of flux components is having a lesser effect up to 1.5-2.0 compositional flux ratios, while after, that it significantly increases the surface tension.
- With an increase of the TiO₂/SiO₂ ratio, the work of adhesion value increased while the maximum value of work of adhesion is obtained in the 1.5 to 1.8 or 2.4 to 2.6 compositional ratio range. With an increase of the TiO₂/MgO ratio, the work of adhesion value firstly increased up to 1.2 to 2.0 flux ratios, but after that, it decreased.

6.1.3 Conclusions from study on Multi-pass bead on plate experimentation for SAW fluxes

- Regression analysis shows that individual flux components gives antisynegistic effect on carbon content for basic flux system while for rutile-basic and rutile-acidic flux system it shows synergistic effect and increases the carbon content. For basic flux system all the binary mixture components decrease the carbon content in multi-pass bead on plate weld deposits experimentation while for rutile-basic flux system binary and ternary mixture has no effect on it. Binary mixture components TiO₂.Al₂O₃ and SiO₂.Al₂O₃ decrease the carbon content and show antisynergistic effect while MgO.Al₂O₃ increases the carbon content and shows synergistic effect. Transfer of carbon from weld to slag is affected by the presence of oxygen in the weld metal.
- Individual flux components significantly increase the silicon content and show synergistic effect for basic and rutile-basic flux system while for rutile-acidic flux system they show antisynergistic effect. CaO.Al₂O₃ is the binary mixture component which increases the weld silicon content for basic flux system while for rutile-basic flux system it decreases. Binary mixture component SiO₂.CaF₂, SiO₂.Al₂O₃ and CaF₂.Al₂O₃ increases it and show positive effect for basic flux system. SiO₂.CaF₂.Al₂O₃ is the only ternary mixture component which significantly increases silicon content for basic flux system. Binary mixture component TiO₂.SiO₂ decreases the silicon content for rutile-basic system while for rutile-cacidic flux system it increases the silicon content. TiO₂.Al₂O₃ increases the silicon content for rutile-basic flux system while for rutile-cacidic flux system it increases the silicon content. TiO₂.Al₂O₃ increases the silicon content for rutile-basic flux system it decreases.
- Regression analysis of manganese shows that all the individual flux components decrease the weld bead manganese content for basic, rutile-basic and rutile-acidic flux systems. SiO₂.CaF₂ is the binary component which increased it for basic flux system. SiO₂.CaO increases the weld bead manganese content while CaO.Al₂O₃ decreases it for rutile-basic flux system. TiO₂.SiO₂ shows positive effect on it while TiO₂.Al₂O₃ and SiO₂.Al₂O₃ show negative effect on manganese content for rutile-acidic flux system. All the ternary mixture component of basic flux system decreases the weld bead manganese content while TiO₂.SiO₂.Al₂O₃ is the only ternary component which shows positive effect on it for rutile-acidic flux system.
- Regression analysis of phosphorous and sulfur shows that all the individual flux components shows antisynergistic effect on weld bead phosphorous and sulphur content for rutile-basic flux system while it shows significant synergistic effect for rutile-acidic flux system. TiO₂.CaO is the binary component which significantly increased its effect for rutile-basic flux system. TiO₂.SiO₂ significantly decreases the weld bead phosphorous content while it increased the weld bead sulphur content for rutile-basic flux system. Binary mixture components TiO₂.Al₂O₃, SiO₂.Al₂O₃ and MgO.Al₂O₃ increase the weld bead phosphorous content for rutile-acidic flux system. All the ternary mixture components decrease the weld bead phosphorous content for basic flux system.
- Regression analysis shows that all the individual flux components decrease the weld bead chromium content for basic and rutile-acidic flux system while for rutile-basic system they tend to increase the chromium content. For basic flux system, all the binary mixture components significantly decrease the chromium content and thus show

antisynergistic effect. Ternary mixture component CaO.CaF₂.Al₂O₃ increases the weld bead chromium content while SiO₂.CaF₂.Al₂O₃ decreases the chromium content. For rutile-acidic flux system, binary mixture components TiO₂.SiO₂ and MgO.Al₂O₃ show positive effect and thus increase the weld bead chromium content while remaining binary component significantly decrease the weld bead chromium content. All ternary mixture components significantly increase the chromium content and thus show synergistic effect on chromium content.

- Regression analysis of molybdenum shows that the individual component decreases the weld bead molybdenum content for basic, rutile-basic and rutile-acidic flux systems. Binary mixture components SiO₂.CaF₂ and CaF₂.Al₂O₃ show antisynergistic effect while CaF₂.Al₂O₃ increases the molybdenum content and thus shows synergistic effect for basic flux system. Ternary mixture of basic flux system decreases the molybdenum content. Binary component CaO.Al₂O₃ shows positive effect for weld bead molybdenum content while SiO₂.Al₂O₃ shows negative effect and thus decreases the molybdenum content. MgO.Al₂O₃ is the only binary component of rutile-acidic flux system which increases the weld bead molybdenum content while all other binary component significantly decreases the molybdenum content.
- Regression analysis of titanium shows that the individual flux component significantly decreases the weld bead titanium content for three flux systems. Binary mixture components CaO.CaF₂, CaF₂.Al₂O₃, TiO₂.CaO and TiO₂.MgO significantly increase the titanium content and thus show synergistic effect for basic, rutile-basic and rutile-acidic flux systems while all remaining binary components decrease the weld bead titanium content for basic and rutile-acidic flux systems.
- Regression analysis shows that linear mixture component decreases the weld bead grain size for basic and rutile-basic flux systems while there is increase in grain size for rutileacidic flux system. TiO₂.SiO₂ is the only binary mixture component which shows antisynergistic effect on grain size while for rutile acidic flux system it shows synergistic effect. Binary mixture component CaO.Al₂O₃ increases the grain size for basic flux system while it decreases the grain size for rutile-basic flux system. Binary mixture component SiO₂.Al₂O₃ significantly increases the grain size for basic system while it decreases the grain size for rutile-acidic flux system. CaO.SiO₂ binary mixture component shows negative effect on weld bead grain size for basic system while it shows positive effect for rutile-basic flux system. For basic flux system other binary components CaO.CaF₂ and CaF₂.Al₂O₃ significantly increase the grain size and thus show synergistic effect while SiO_2 . CaF₂ shows antisynergistic effect. Binary component TiO₂.SiO₂ shows negative effect on grain size for rutile-basic flux system while it shows positive effect on grain size for rutile-acidic flux system. Binary mixture component TiO₂.CaO, TiO₂.MgO, TiO₂.Al₂O₃ and MgO.Al₂O₃ significantly decrease the grain size for rutile-basic and rutile-acidic flux system. TiO2.SiO2.CaO is the only ternary mixture component which significantly increases the grain size for rutile-basic flux system.
- Regression analysis of microhardness shows that individual flux component decreases the weld bead microhardness for basic flux system while it increases the microhardness for rutile-basic and rutile-acidic flux system and shows synergistic effect. SiO₂.Al₂O₃ is the only binary mixture component which significantly increases the weld bead microhardness for basic as well as rutile-basic flux system. Binary mixture components CaO.Al₂O₃, CaF₂.Al₂O₃ and TiO₂.Al₂O₃ show significant synergistic effect on weld bead microhardness for basic and rutile-basic flux system while CaO.SiO₂, CaO.CaF₂ and SiO₂.CaF₂ show antisynergistic effect on microhardness.

6.1.4 Conclusions from corrosion study of base metal

The effects of heat treatment (HT-1 and HT-2) on corrosion rate due to weight loss, mechanical and microstructutal behaviour of API X70 pipeline steel in four different exposing environments were investigated.

- Corrosion rate is higher in sea water as compared to fresh water. In sea water study maximum corrosion rate (0.489 mm/y) was observed for HT-1 specimens tempered at 600°C followed by HT-2 specimens tempered at 450°C. Minimum corrosion rate (0.072 & 0.052 mm/y) was observed for heat treated HT-1 and HT-2 specimen tempered at 300°C.
- In sea water study, maximum average microhardness (218.81HV) was observed for HT-2 specimens tempered at 300°C due to the presence of tempered martensite phase in the microstructure while minimum value of average microhardness was observed for HT-1 specimen tempered at 450°C due to the presence of fine ferrite plus bainite phase in the microstructure.
- For heat treated specimen, maximum impact toughness was observed for HT-1 (438 J) specimen tempered at 600°C while HT-2 specimen tempered at 450°C shows least impact toughness value.
- In fresh water study, maximum corrosion rate (1.708 mm/y) was observed for HT-2 specimen tempered at 300°C while minimum corrosion rate was observed (0.177 mm/y) for HT-1 specimen tempered at 600°C.
- Maximum microhardness (191.40 HV) was observed for HT-1 specimen tempered at 450°C in fresh water while minimum average microhardness (155.54 HV) was observed for HT-2 specimen tempered at 450°C. Low value of average micohardness is generally due to the presence of lamellar ferrite as well as precipitates of pearlite in the microstruture.
- For sodium thiosulphate medium, maximum value of corrosion rate (0.489 mm/y) was observed for HT-2 specimen (tempered at 300°C) immersed in 5%NaCl+10⁻² mol/l (pH=3) solution while lowest value of corrosion rate (0.329 mm/y) was observed for HT-1 specimen (tempered at 450°C) immersed in 5%NaCl+10⁻² mol/l (pH=3) medium.
- Heat treated specimen (HT-1) immersed in 5%NaCl+10-2mol/l sodium thio-sulphate solution, pH=3 (tempered at 600°C) shows maximum value of average microhardness (231.52 HV) while HT-1 specimen tempered at 450°C immersed in same medium results in lower value of average microhardness (197.23 HV). HT-2 specimen immersed in 5%NaCl+10-2mol/l sodium thio-sulphate solution, pH=3 (tempered at 450°C) shows maximum value of average microhardness while HT-2 specimen tempered at 600°C immersed in same medium results in lower value of average microhardness while HT-2 specimen tempered at 600°C immersed in same medium results in lower value of average microhardness (149.91).
- Heat treated specimen (HT-1) immersed in 5%NaCl+10⁻³mol/l sodium thio-sulphate solution, pH=5 (tempered at 450°C) shows maximum value of average microhardness (215.22 HV) while HT-1 specimens tempered at 600°C immersed in same medium results in lower value of average microhardness (198.23 HV) just opposite to that observed in 5%NaCl+10⁻²mol/l sodium thio-sulphate solution, pH=3. This is due to higher dissolution or more precipitation of solid sulphide ions on the metal surface in more acidic medium (pH=3). This (more acidic solution, pH=3) may reduce the strengthening bond between fine grains and results in lower value of hardness. HT-2 specimen immersed in 5%NaCl+10⁻³mol/l sodium thio-sulphate solution, pH=5 (tempered at 450°C) shows maximum value of average microhardness (215.22 HV) while HT-2 specimen tempered at 600°C immersed in same medium results lower value of average microhardness (144.51 HV).

The effects of heat treatment (HT-1 and HT-2) on electrochemical corrosion and microstructural behaviour of API X70 pipeline steel in sea water, sodium thiosulphate solution, pH=3 was also investigated.

- Maximum corrosion rate (0.065218 mm/y) was observed for base metal when exposed in 5%NaCl+10⁻²mol/l sodium thio-sulphate solution, pH=3 as compared to sea water and 5%NaCl+10⁻³ mol/l sodium thio-sulphate solution, pH=5. This is because in highly acidic medium (pH=3) hydrogen sulphide quickly accelerate the cathodic as well as anodic reactions of the corrosion process i.e. H₂ evolution reactions or iron dissolution. HT-1 specimen tempered at 600° C shows maximum corrosion rate in sea water medium while specimen tempered at 300° C and 450° C show almost similar corrosion behaviour. HT-2 specimen tempered at 450° C show maximum corrosion rate (0.025700 mm/y) in sea water solution as compared to specimen tempered at 300° C.
- HT-1 specimen tempered at 300° C show maximum corrosion rate (0.094099 mm/y) in 5%NaCl+10⁻²mol/l sodium thio-sulphate solution, pH=5 as compared to specimen tempered at 450° C and 600° C. Tempered martensite microstructure was observed for HT-1 specimen tempered at 300° C. The ease of diffusion of corrosion products (e.g. hydrogen sulphide ions) is more prone in coarse grained microstructure. But opposite results were observed for HT-2 specimen exposed in the same medium. HT-2 specimen tempered at 450° C and 300° C show lower corrosion rate as compared to 600° C tempered specimen.

6.1.5 Conclusion from study of weld metal

The mechanical properties (impact toughness, tensile strength and microhardness), chemical composition, microstructure and hydrogen induced cracking (HIC) behaviour of API X70 pipeline steel welds was studied (using commercial as well as laboratory prepared agglomerated fluxes). The tested weld specimen has boron, niobium, manganese, molybdenum, chromium, copper and titanium as the primary alloying elements.

- It was observed that Ni and Nb content in the weld metal decreased while Ti, B, Cu and Cr (carbide former) content significantly increased in the fusion zone as compared to the base metal composition.
- Sulphur content significantly increased for all the weld joints (as compared to BM) while a maximum increase was observed for weld specimen F19RA and the minimum increase was seen for specimen F5B.
- Base metal, C.F weld joint (commercial flux) and F5B weld specimen have a similar value of carbon equivalent and possess good weldability as compared to the other weld specimen. Weld specimen F7RB, F5RB, F19RA and F3RA show a lower value of carbon equivalent.
- C.F (commercial flux) weld specimen show maximum tensile strength (613 N/mm²) value as compared to all the other weld specimen except base metal. A higher value of strength for commercial joint is attributed to the larger value of carbon equivalent. Weld specimen prepared by using basic flux F15B shows the maximum tensile strength (561.8 N/mm²).
- Weld specimen F15B shows the maximum percentage elongation of 23.57% as compared to all the remaining weld joints including the commercial flux weld joint (22.8%).
- Weld joint prepared by using acidic flux (i.e. F3RA and F19RA) shows poor impact toughness properties. This is due to the presence of more acidic content (more oxygen content) in the weld region. Presence of higher oxide inclusion in the weld region exhibit poor impact toughness properties.
- Maximum microhardness of 230 HV and the minimum of 190 HV was observed for weld metal and it is similar to the base metal microhardness (240 HV & 185 HV). This may be due to the presence of lower temperature transformation products in the weld zone. Both C.F and F15B weld joints have almost similar microhardness value in the weld

zone. All weld specimen shows good combination of strength and toughness value and fulfill the API service requirements.

- Metallographic examination of all the weld specimen show blend of ferrite-bainite (base metal), acicular ferrite (weld region), polygonal-ferrite (weld region) with dispersed carbides, polygonal-ferrite plus windanstten-ferrite (weld region), tampered martensite (HAZ), coarse-grained heat-affected zone (CGHAZ) and fine-grained heat-affected zone (FGHAZ).
- Weld specimen F3RA and F19RA shows maximum crack sensitivity ratio (CSR %) in hydrogen induced cracking measurements. Base metal, F5B and C.F weld specimen shows lower CSR (Crack sensitivity ratio) as compared to the remaining weld specimen.

6.2 SCOPE FOR FUTURE WORK

More ternary phase systems can be explored for the development of submerged arc fluxes for welding of higher API pipeline grades. Use of nano powder addition and recyclable slag can be explored while developing submerged arc welding fluxes. In present work, the regression models using physicochemical and thermo-physical properties such as density, gain fineness number, percentage weight change, enthalpy, thermal conductivity, thermal diffusivity and specific heat for three submerged arc welding flux systems were developed for welding of API X70 pipeline steel. Weld bead chemistry, average grain size and microhardness in multi-pass bead on plate weld deposit have been analyzed for three different flux systems. Mechanical properties such as tensile strength, impact toughness, microhardness and hydrogen induced cracking of seven weld specimen (using laboratory developed fluxes) were studied.

Further scope includes the mechanical tests such as fatigue testing under different loading conditions, measurent of weld metal hydrogen content, corrosion and hydrogen induced cracking behaviour of different pipeline steel grades (e.g. X80, X100 X120 etc.) in different exposing environments and comparison with that of commercially available SAW fluxes.