

Kinetic And Correlation Studies Between Varying Corrosion Parameters And Corrosion Rate Of N-80 Oil Well Tubular Steel In 15% Hydrochloric Acid Pickling Solution Using *Ficus Abutilifolia* Plant Extract

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ABSTRACT

The varying effects of temperature, time and inhibitor concentrations on the corrosion rate of N-80 oil well tubular steel in 15% hydrochloric acid with and without leave extract of *Ficus Abutilifolia* have been investigated. The time, temperature and concentration of inhibitor were varied in the range 1-2.5 hrs at 0.5 hr interval, 45°C-90°C at 15°C interval and 0.1 – 0.9 v/v% at 0.1v/v% interval respectively. Standard deviation, effect of variables at 95% confidence interval, factorial design, and Arrhenius kinetics approach were used to correlate the corrosion parameters and corrosion rate. The obtained results indicate that temperature increase enhances corrosion rate while increase in inhibitor concentration and time decreases corrosion rate. It was found also that temperature and inhibitor concentration are the most important control parameters for corrosion process. The kinetic and statistical approaches were in good agreement.

Keywords: Corrosion inhibition, *Ficus Abutilifolia*, hydrochloric acid, kinetic and statistical analysis.

1. INTRODUCTION

Corrosion of metals is a major industrial problem that has attracted a lot of investigators in recent years (Liu *et al*, 2001; Ita and Offiong, 1999; Ekpe *et al*, 1995; Collins *et al*, 1993). In industries such as pulp and paper industry, power generation, underground structures, chemical and oil industries, metals are used in over 90% of construction process (Osarolube *et al.*, 2004). Iron and steel are the most commonly used materials in the fabrication and manufacturing of oil field operating platforms because of their availability, low cost, ease of fabrication, and high strength (Umezurike, 1998; Nwoko and Umoru, 1998). Most industrial media are usually rich in elemental gases, inorganic salts, and acidic solutions most of which influence corrosion rates, and mechanisms (Owate and Abumere, 2003; Abiola and Oforka, 2005). The highly corrosive nature of aqueous mineral acid used with most engineering materials found in industrial environment requires some degree of restraint to achieve economic maintenance and operation of equipments or materials and maximum safety conditions (Kuznetsov and Yu, 2002). Metals are usually exposed to the action of bases or acids in the industries. The most important industrial fields of application are acid pickling, industrial acid cleaning, acid descaling and oil well acidification (Hosseini *et al*, 2003a; Fan *et al*, 2002; Hosseini *et al*, 2003b).

During pickling, various hot acid solutions are used for removing mill scales (oxide scales) from the metal surface at elevated temperatures, such as 60°C in hydrochloric acid and up to 95°C in sulfuric acid (Sadaf and Quraishi, 2010). Thus in this work maximum temperature of 90°C was used.

Corrosion prevention of metal based on the use of corrosion inhibitors which when present in a system at sufficient concentrations, decrease the corrosion rate without causing considerable changes in the concentration of any corrosion reagent (standart ISO 8044, 1989). Considering that losses from metal corrosion exceed 5% of the gross national product in industrial countries, the development and application of inhibitors, which is one of the main methods of corrosion protection, should be considered as a scientific and engineering task of current interest. Studies have revealed that the existence of various inhibitors namely oxidizing, adsorption, complexation-type and polymeric type (Kuznetsov and Yu, 2002).

The best known oxidizing inhibitors are the salts of chromic acid. However, discharge of chromate has become unacceptable, due to environmental hazards.

The recent trend is towards environment – friendly inhibitors. Most of the natural products are non-toxic, biodegradable and readily available in plenty. Various parts of plants – seeds, fruits, leaves, flowers, etc – have been used as corrosion inhibitors (Rajendran, *et al*, 2005).

The cost of using green inhibitors is less when compared to that of organic inhibitors which takes lot of chemicals and also time for its preparation (Sharmila *et al*, 2010).

Thermodynamic and kinetic parameters are used for analyzing systems undergoing chemical reactivity. However, corroding systems are not in equilibrium and therefore thermodynamic calculation cannot be applied. Hence from the engineering point of view, the major study of interest is the kinetic rate of corrosion. Despite their abundance in our environment no previous work has been undertaken on the corrosion inhibition properties of *Ficus Abutilifolia*. Therefore, investigation into its corrosion inhibition properties will provide pioneering knowledge on the use of its leave extract as inhibitors and therefore add to the growing list of relatively cheap, environmentally friendly and non-toxic corrosion inhibitors. Hence in the present work, the extract of *Ficus Abutilifolia* leaves is evaluated for its corrosion inhibition performance by varying its corrosion parameters using Weight loss method.

2. Materials and Methods

2.1. Materials

The experimental materials used in this study are: Plant extract, Hydrochloric Acid (Analar Grade), Ammonium Acetate (Analar Grade), Distilled Water, Emery papers (220-800 grades), 250ml beakers, retort stand, N-80 steel coupons.

Table 1 The high strength low alloy steel is a commercial micro alloyed steel (in hot-rolled condition) with the following percentage alloy

| Element | C | Mn | Si | P | S | Mo | V | Ti | Al | Fe | Total |
|---------------|------|-----|------|-------|-------|-------|-------|-------|-------|-------|-------|
| Composition,% | 0.22 | 1.2 | 0.25 | 0.025 | 0.025 | 0.001 | 0.004 | 0.002 | 0.051 | 98.22 | 100 |

2.2. Equipment

The equipments used in this study are as follows: thermostated water bath, analytical balance.

2.3. Determination of corrosion rate

The as – received steel (6cm x 5cm x 1.5cm) bars were sectioned into coupon of sizes 2cm x 1cm x 0.75cm and polished with emery papers (220-800 grades).

The specimens were the washed with distilled water followed by degreasing in absolute ethanol, dried in acetone, weighed in an analytical balance and stored in a moisture free desiccators prior to use ^[16]. Corrosion tests were carried out in freely aerated freshly prepared 15% hydrochloric solution. This was prepared from AR grade 36% HCl and distilled water. Freshly prepared extract of *Ficus Abutilifolia* were added at concentrations of 0.1 v/v%, 0.3 v/v%, 0.5 v/v%, 0.7 v/v% and 0.9 v/v%. All the experiments were conducted at 45°C, 60°C, 75°C and 90°C. The total immersion time was 150 minutes while the corrosion rate was examined every 30minutes, to ascertain the effect of time on the corrosion rate.

3. Results and discussion

3.1. Results

3.1.1. Gravimetric test

To compare the factors that influence corrosion rate of the N-80 Alloy steel, the corrosion rates of the experimental specimens immersed in the corrodent with and without inhibitor at varied temperature and time were determined using the standard expression for measurement of Corrosion rate in mils per year (mpy) which is expressed in the equation.

$$r = 534W/\rho At \text{ -----1}$$

where r is the corrosion rate, W is the weight loss (mg), ρ is the density of the material (g/cm³), t is the time of exposure (hrs) and A is the total surface area (in²). The results obtained are shown in Figures 1-5.

3.1.2. Kinetic analysis

Kinetic models are a useful tool to explain the mechanism of corrosion inhibition processes. Arrhenius equation was used to determine the corrosion kinetic using the expression (Oloche *et al*, 2009).

$$r = B_o e^{-E_a/RT} \text{ -----2}$$

The natural logarithm of the corrosion rate, r, could be represented as a straight line function of 1/T of steel in acid medium (Tang *et al*, 2003; Bouklah *et al*, 2005).

$$\ln r = E_a/RT + \ln B_o \text{ -----3}$$

Where E_a represents the activation energy, R is the universal gas constant, T is the absolute temperature and B_o is the pre-exponential factor. A plot of ln r against 1/T for a range of temperatures gives a linear graph with slope -E_a/R. Hence the apparent activation energy Ea was calculated and tabulated in Table A.1. Figures 6 and 7 show the linear regression of ln r and 1/T from blank (0 v/v%) to 0.9 v/v% concentration of the inhibitor. Figures 8 and 9 show the plots of apparent activation energy as a function of concentration and time.

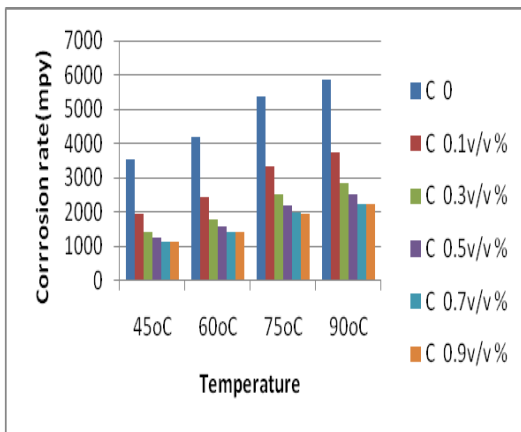


Fig. 1: Variation of corrosion rate with temperature after 0.5 hr exposure time

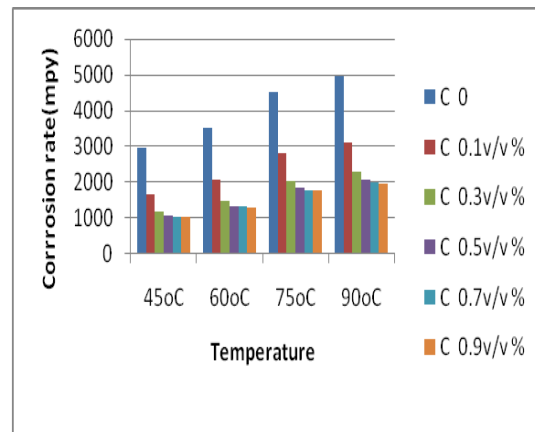


Fig. 2: Variation of corrosion rate with temperature after 1 hr exposure time

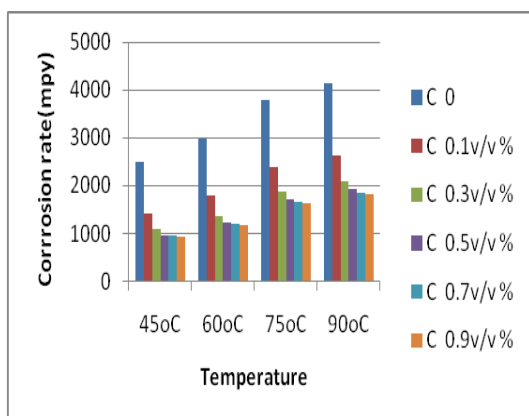


Fig. 3: Variation of corrosion rate with temperature after 1.5 hr exposure time

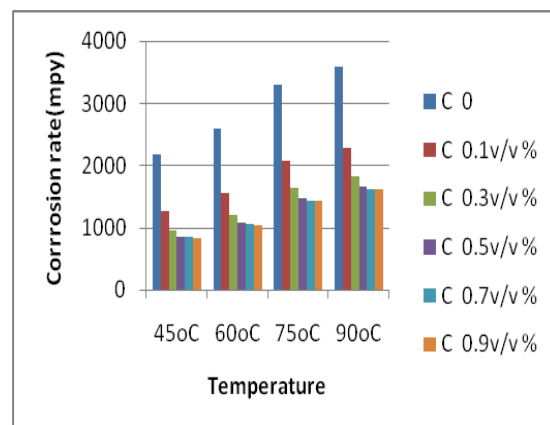


Fig. 4: Variation of corrosion rate with temperature after 2 hrs exposure time

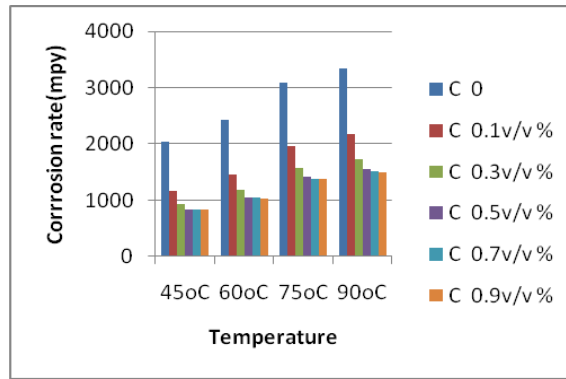


Fig. 5: Variation of corrosion rate with temperature after 2.5 hrs

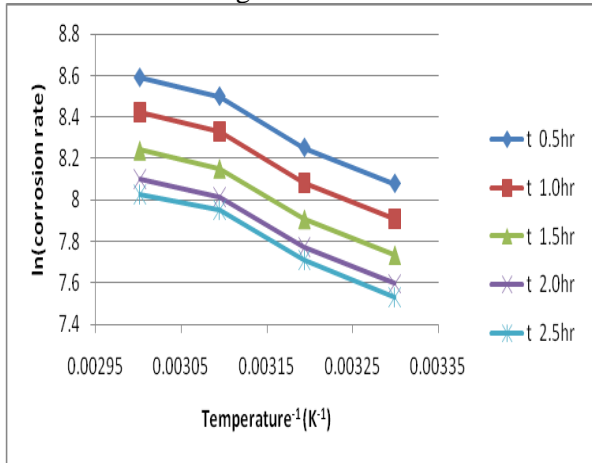


Fig. 6: Variation of ln(corrosion rate) with absolute temperature in HCl with FA extract

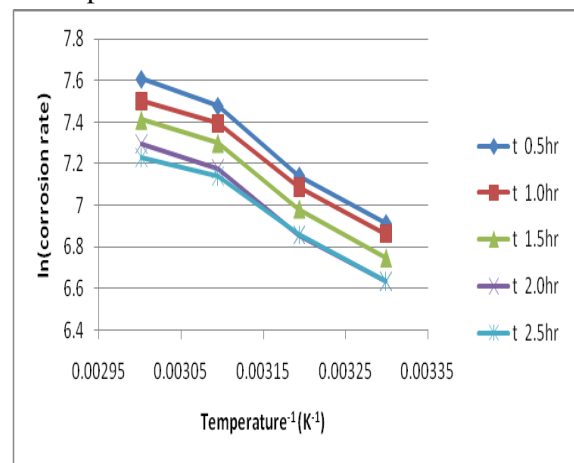


Fig. 7: Variation of ln(corrosion rate) with reciprocal of absolute temperature in HCl without inhibitor

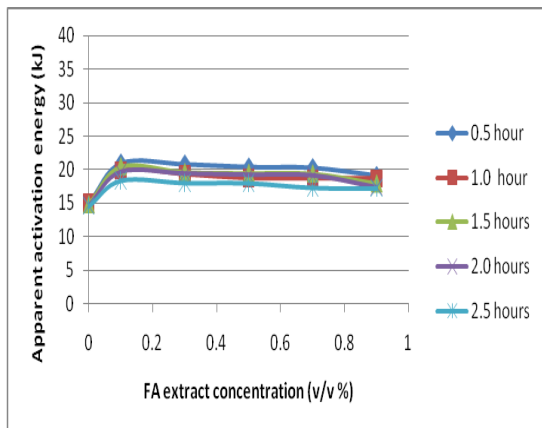


Fig. 8: Variation of apparent activation energy (E_a) with concentration of FA extract (v/v %)

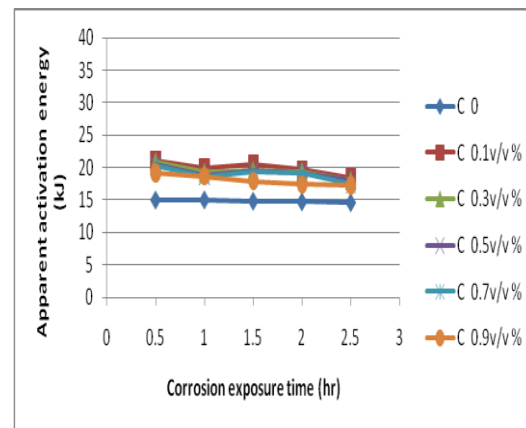


Fig. 9: Variation of apparent activation energy (E_a) with corrosion exposure time (hr)

3.1.3. Statistical analysis of corrosion inhibition process

Two levels, three factors factorial design were used for the statistical analysis (Miller and Freund, 1987, Duncan and Irwin, 1965). The levels for the three factors are entered in Table A2 and the treatment for the two levels and these three factors are tabulated on Table A3.

The significant factors (main and the interactions) were identified by Yates analysis (Miller and Freund, 1987).

The Yates analysis computation for the effects is shown in Table A5. The standard deviation of the effects was also calculated. The results are shown in Table A6. The 95% confidence interval for the estimated effect is given as (Oloche *et al*, 2009).

$$E \pm (t_{\infty, 0.05}) \times (\text{standard deviation of the estimated effect}) \text{-----}4$$

Where $(t_{\infty, 0.05})$ is 2.31. The results of the estimated effect at 95% confidence interval are shown in Table A7.

3.2. Discussion

Figures 1-5 indicate that the corrosion rate increases with temperature and decreases with inhibitor concentration. The figures show a change in corrosion rate of 2350 mpy, 1987 mpy, 1643 mpy, 1419 mpy and 1313 mpy of the specimen immersed in the absence of inhibitor when the process experimental temperature was raised from 45 °C to 90°C at an exposure time of 0.5, 1.0, 1.5, 2.0 and 2.5 hours respectively. The figures also show a change in corrosion rate of 831 mpy, 610 mpy, 491 mpy, 428 mpy and 329 mpy at an experimental temperature of 45°C when the inhibitor concentration was increased from 0.1 v/v% to 0.9 v/v% at an exposure time of 0.5, 1.0, 1.5, 2.0 and 2.5 hrs respectively.

However, at all combinations of temperature and inhibitor concentration, the corrosion rates decrease with increase in exposure time. Hence, temperature and inhibitor concentration are the most significant parameters for corrosion rate control.

Figures 6 and 7 represent the linear regression of $\ln r$ and $1/T$ for the specimen immersed in 15% HCl with and without corrosion inhibitor. Some studies (Zhao and Mu, 1999; Khamis, 1990) show that compared with the apparent activation energy in the absence of inhibitor, higher values of E_a were found in the presence of inhibitors. This suggests existence of higher energy barrier for the corrosion process in the inhibited solutions associated with physical adsorption or weak chemical bonding between the inhibitor species and the steel surface (Li and Mu, 2005). Other studies show that in the presence of inhibitors, the apparent activation energy was lower than that in the absence of inhibitor (Banerjee and Misra, 1989; Cai *et al*, 1993). It could be found from Figure 8 that the apparent activation energy for the entire specimen exposed to 15% HCl solution was higher than that without inhibitors (Figure 7), which suggests that *Ficus Abutilifolia* extract is an active inhibitor of the steel corrosion. Similar observation has been reported (Oloche *et al*, 2009).

Activation energy was however greatest with inhibitor addition of 0.1 v/v% at all the test durations. However, E_a values decrease as concentration is increased beyond 0.1 v/v%. The decrease in the E_a value as observed for the inhibitors at higher inhibitor concentration has been reported in the literature (Li, X and Mu, G, 2005; Tang *et al*, 2006; Tang *et al*, 2003; Obot and Obi-Egbedi 2009; Ebenso *et al*, 2009). Riggs and Hurd (1967) explained this observation by stating that at higher levels of inhibition the net corrosion reaction shifts from that on the uncovered part of the metal surface to the covered one. This trend also suggests that FA is an active inhibitor on steel in 15% HCl solution (Oloche *et al*, 2009 and Ebenso, 2003). The increase in value of activation energy in the presence of FA signify physical adsorption. The values of E_a obtained for all the inhibitors indicated that the whole process is controlled by surface reaction, since the activation energies of the corrosion process is over 20 kJ/mol (Al-Neami *et al*, 1995). A comparison of Figures 8 and 9 shows a steeper slope in Figure 8 (E_a against concentration of corrosion inhibitor), than the almost horizontal lines of Figure 9 (E_a against time). This observation indicates that change in inhibitor concentration has more effect on the corrosion rate than exposure time. Hence, temperature and inhibitor concentration are the most important kinetic parameters in the corrosion inhibition of steel in 15% HCl with FA as corrosion inhibitor.

From the results of the factorial design for FA (Table A6), the experimental temperature appears to be the most important variable with main effect of 1047.3 mpy followed by concentration with -767.3 mpy and time -748.8 mpy. The analysis shows that raising the temperature from 45°C to 90°C would result in increase in corrosion rate by 1047.3 mpy. Increasing concentration from 0.1 v/v% to 0.9 v/v% would decrease corrosion rate by 767.3 mpy while increasing time from 0.5 to 2.5hrs would decrease the corrosion rate by 748.8 mpy.

The estimated interactions between temperature and concentration, temperature and time, concentration and time and then between all three factors temperature, concentration and time are -235.2 mpy, -273.2 mpy, 308.6 mpy and 77.9 mpy respectively. Of all the interactions, concentration and time is most significant. Raising the two at the same time leads to increase in corrosion rate by 308.6 mpy while raising temperature and time would result in decrease in corrosion rate by 273.2 mpy. Similarly, increasing temperature and concentration would result in increase in corrosion rate by 235.2 mpy while increasing all three factors simultaneously increases corrosion rate by 77.9 mpy. The result in Table A7 shows values for all the main effects of the factors with temperature being the highest with a value of 4.32 times the standard deviation value. This again shows that temperature is the most significant parameter in tubular steel corrosion control using extract of FA as inhibitor. From the results of Table A8, temperature and inhibitor concentration are the most significant parameters in corrosion inhibition of tubular steel at 95% confidence level. The results therefore implies that the results of the statistical analysis comprising Yates factorial design, standard deviation

and effect of variables at 95% confidence level, confirm kinetic (activation energy) result that temperature and inhibitor concentration are the most important parameters in the inhibition of tubular steel.

4. Conclusions

1. Extract of *Ficus Abutilifolia* significantly reduced the corrosion rate of tubular steel in 15% HCl solution.
2. the corrosion rate increases with temperature and decreases with inhibitor concentration
3. Maximum corrosion inhibition was observed at inhibitor concentration of 0.9 v/v%.
4. The values E_a for all the inhibitors studied were all less 80KJ/mol indicating physical adsorption mechanism.
5. Yates factorial design, standard deviation and effect of variables at 95% confidence level, confirm that temperature and inhibitor concentration are the most important parameters in the inhibition of tubular steel.
6. Kinetic and statistical analysis are in agreement, confirming that temperature and concentration are the most important corrosion inhibition parameters.

Table A1 Results of apparent activation energy with and without inhibitor at different exposure time

| Inhibitor concentration(v/v%) | Apparent activation energy (kJ/mol) at different times (hr) | | | | |
|-------------------------------|---|--------|--------|--------|--------|
| | 0.5 hr | 1.0 hr | 1.5 hr | 2.0 hr | 2.5 hr |
| 0 | 15.02 | 15.02 | 14.84 | 14.75 | 14.61 |
| 0.1 | 21.01 | 19.89 | 20.46 | 19.66 | 18.42 |
| 0.3 | 20.85 | 19.28 | 19.60 | 19.39 | 18.08 |
| 0.5 | 20.43 | 18.69 | 19.47 | 19.22 | 18.04 |
| 0.7 | 20.33 | 18.68 | 19.38 | 19.13 | 17.36 |
| 0.9 | 19.16 | 18.66 | 17.91 | 17.39 | 17.25 |

Table A2: Factorial design of the corrosion rate

| Factor | Low level | High level |
|-------------------------|-----------|------------|
| Temperature, T (°C) | 45 | 90 |
| Concentration, C (v/v%) | 0.1 | 0.9 |
| Time, t (hr) | 0.5 | 2.5 |

Table A3: Factorial design of the corrosion process showing the treatment combination

| Exp.number | Temperature level | Concentration level | Time level |
|------------|-------------------|---------------------|------------|
| 1 | -1 | -1 | -1 |
| T | +1 | -1 | -1 |
| C | -1 | +1 | -1 |
| TC | +1 | +1 | -1 |
| t | -1 | -1 | +1 |
| Tt | +1 | -1 | +1 |
| Ct | -1 | +1 | +1 |
| TCt | +1 | +1 | +1 |

Coded: -1(low level),+1(high level)

Table A4: Experimental Condition and Corrosion Rate for Each Condition for FA

| Experimental combination | Temp(°C) | Conc(v/v%) |
|--------------------------|----------|------------|
| 1 | 45 | 0.1 |
| T | 90 | 0.1 |
| C | 45 | 0.9 |
| TC | 90 | 0.9 |
| T | 45 | 0.1 |
| Tt | 90 | 0.1 |
| Ct | 45 | 0.9 |
| TCt | 90 | 0.9 |

Table A5: Yates Analysis

| T.combn | CR | Col 3 | Col 4 | Col 5 | ME |
|---------|----------|---------|--------|---------|---------|
| I | 1768.76 | 5171.19 | 8190.5 | 13385.7 | 3346.43 |
| T | 3402.436 | 3019.30 | 5195.2 | 4189.10 | 1047.27 |
| C | 1005.9 | 3056.35 | 2640.9 | -3069.3 | -767.34 |
| TC | 2013.31 | 2138.87 | 1548.1 | -940.8 | -235.22 |
| t | 1062.51 | 1633.6 | -2151 | -2995.2 | -748.81 |
| Tt | 1993.84 | 1007.3 | -917.4 | -1092.8 | -273.21 |
| Ct | 761.042 | 931.32 | -626.3 | 1234.4 | 308.60 |
| TCt | 1377.83 | 616.78 | -314.5 | 311.81 | 77.95 |

T.combn=treatment combination

ME=Mean effect

Table A6: Calculation of the Standard deviation of the effects

| Y | Y-S | [Y-S] ² |
|---|----------|--|
| 1768.763 | -95.547 | 9129.229 |
| 3402.436 | -1729.22 | 2990202 |
| 1005.993 | 667.223 | 445186.5 |
| 2013.31 | -340.094 | 115663.9 |
| 1062.514 | 610.702 | 372956.9 |
| 1993.843 | -320.627 | 102801.7 |
| 761.0422 | 912.1738 | 832061 |
| 1377.83 | 295.386 | 87252.89 |
| $\frac{\sum Y}{N} = \frac{13385.7}{8} = 1673.2$ | | $S = \frac{\sum [Y - S]^2}{N} = \sqrt{\frac{4955254}{8}} = 787.02$ |

S = Standard deviation, Y=Effect, N=Number of effects

Table A7: Effects of variables at 95% Confidence level

| Main effect | Effect±(t _{α,0.05})*S | 95% Confidence interval |
|--------------------------|---------------------------------|-------------------------|
| T | 1047.28±1818.03 | -770.75+2865.31 |
| C | -767.35±1818.03 | -2585.38+1050.68 |
| T | -748.82±1818.03 | -2566.85+1069.21 |
| Two-factor interaction | | |
| TC | -235.22±1818.03 | -2053.25+1582.81 |
| Tt | -273.22±1818.03 | -2091.25+1544.81 |
| Ct | 308.60±1818.03 | -1509.43+2126.63 |
| Three-factor interaction | | |
| TCt | 77.95±1818.03 | -1740.08+1895.98 |

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