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Eldewaik, Marwa; Gadalla, Mamdouh A. Prof.; Radwan, M A.; Sadek, M.A; and Elazab, Hany A., "The Kinetic Study of DPT using Mathematica as an Efficient Optimization Tool" (2020). *Basic Science Engineering*. 6. https://buescholar.bue.edu.eg/basic_sci_eng/6

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The Kinetic Study of DPT Using Mathematica as an Efficient Optimization Tool

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ABSTRACT

Mathematica is a powerful program for computing both numeric and algebraic calculations as well as graphing two and three dimensional curves and surfaces. It is used increasingly in many fields of science now such as physics, engineering, chemistry and even biology because of the fast interaction of mathematics with almost the fields of science nowadays. Synthesis of Cyclotetramethylene Tetramine through the action of nitrating mixture formed of ammonium nitrate and fuming nitric acid on hexamine in presence of acetic acid, acetic anhydride and p-formaldehyde has been proven. The pathway is relatively long and Hexamine Dinitrate and Dinitro Pentamethylene Tetramine (DPT) are two of the main intermediate compounds. The former was prepared, purified, and then characterized. Conversion of this compound into the latter has been followed up experimentally. Herein, we report the Preparation of Dinitro Pentamethylene Tetramine (DPT) from Hexamine Dinitrate - as an alternative synthetic route - which is an important intermediate appears through preparation of DPT from Hexamine directly. DPT was prepared at different temperatures. The variation of some factors like: temperature and time has been investigated. The obtained results were reliable and consistent with the literature. The conversion of Hexamine Dinitrate to HMX as another synthetic route was not fully studied from the point of view of kinetics. In this paper, it is intended to study the effect of time and temperature on the conversion rate of hexamine dinitrate to DPT. This scientific approach is considered as a bridge through which we aim to initiate a complete kinetic study of an important intermediate in the synthesis route of one of the most powerful energetic materials.

We report here, optimizing the kinetic data for the synthesis of DPT via using Mathematica.

Key words: Mathematica, DPT; Kinetics, Optimization.

1.INTRODUCTION

Energetic materials such as explosives, propellants and pyrotechnics are widely used for both civilian and military applications. Design of future defense systems requires the use of energetic material formulations having enhanced performance (energy output) and reduced vulnerability during manufacturing, handling, storage and transportation. Several important design considerations for such formulations include improved mechanical properties, extended service life and reduced environmental impact in manufacture, use and disposal [1]. Energetic materials are substances or mixtures that react chemically to release energy required for their intended application.

Bachmann and Sheehan [2] developed a method of preparing DPT. This method involved nitrolysis of hexamine with ammonium nitrate – nitric acid solution and acetic anhydride.. [1-19]

Hexamine Dinitrate is an important raw material in production of DPT, hexogen (RDX) and HMX as a source of the methylene base and the simultaneously bound “anhydrous” nitric acid in an advantageous crystalline and relatively stable form [20-28]

HMX is considered as a powerful military explosive, but it has a limited use due to its high cost. Reported manufacturing processes for HMX involves the nitration of hexamine. It is well known that HMX can be prepared from DPT, which has been identified as a key intermediate in the formation of HMX from the nitration of hexamine, which is also known as the modified Bachmann process as previously mentioned.

The alteration of those previously mentioned parameters will lead to optimized process and hence obtain better results.[29-37]

Direct nitrolysis of 3,7-dinitro-1,3,5,7-tetraazabicyclo[38-45] nonane (DPT) is a feasible way to synthesize HMX, and it has multiple practical applications. A new nitrolysis process involving the use of an N₂O₅-HNO₃ system catalyzed by acidic ionic liquids (AILs) was developed. The effect of metallic ions on the nitrolysis of DAPT [3,7-diacetyl-1,3,5,7-tetraazabicyclo(3.3.1) nonane] and HA

(hexamine) was investigated by experimental and theoretical approaches [45-69]

2. EXPERIMENTAL

Samples were prepared and filtered in a special fuming hood equipped with glass shelter and a powerful air suction system for ventilation. Safety regulations have been strictly applied. The preparation setup consisted of a flat-bottom 0.5L flask equipped with a mechanical stirrer, three dropping funnels, and a thermometer. Filtration of the prepared samples was done using Buchner funnel - pump system. To handle these kinds of energetic materials, best safety practices (leather gloves, face shield) are strongly recommended. [45]

DPT was also prepared starting from the hexamine dinitrate. To a mixture formed of glacial acetic acid (5ml, 0.0874mol) and acetic anhydride (2ml, 0.0212mol), hexamine dinitrate (1g, 0.00375mol) was added as one portion. Then the reaction mixture was left, for a fifteen minute period, at a temperature of $(44 \pm 1^\circ \text{C})$ which was also maintained throughout this procedure. The reaction mixture was then quenched by chilling it to 12°C . Rapid separation of the solid phase was then carried out.

3. RESULTS AND DISCUSSION

Mathematica is used to obtain more accurate and optimized results.

Table 1: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 15°C .

Time (min.)	0	5	10	15	120	240	360	480	600
Hexamine dinitrate (mg / ml)	0.592	0.589	0.587	0.583	0.541	0.480	0.44791	0.385	0.355
DPT (mg / ml)	0	0.001	0.002	0.005	0.032	0.072	0.10200	0.126	0.135

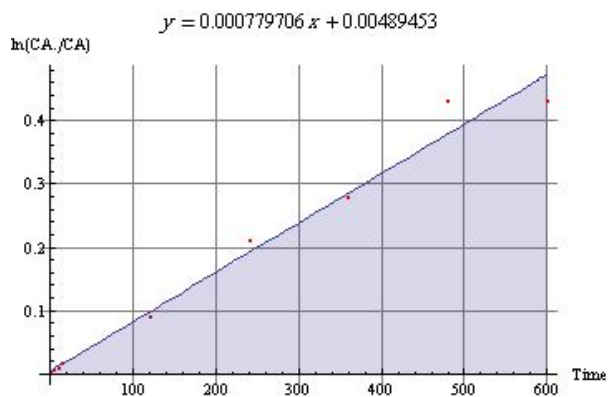


Figure. 1: Representation of the hexamine dinitrate concentration-time data at 15°C - First Order, $R^2=0.997832$.

The arte constant is simply calculated from the obtained concentration results as shown in Figure 1 based on the calculated results from Table 1. [45]

The reaction is generally very slow. 600 minutes were sufficient to convert about 40% only from hexamine dinitrate into DPT as shown in Table 1.

$$y = 0.00183882x + 1.66674$$

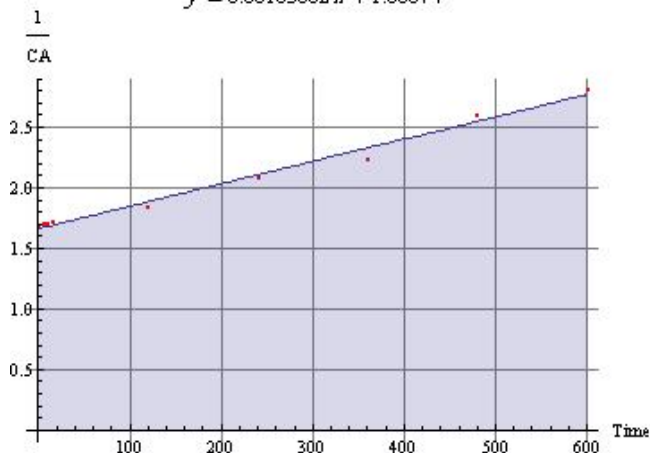


Figure 2: Representation of the hexamine dinitrate concentration-time data at 15°C - Second Order, $R^2=0.994147$.

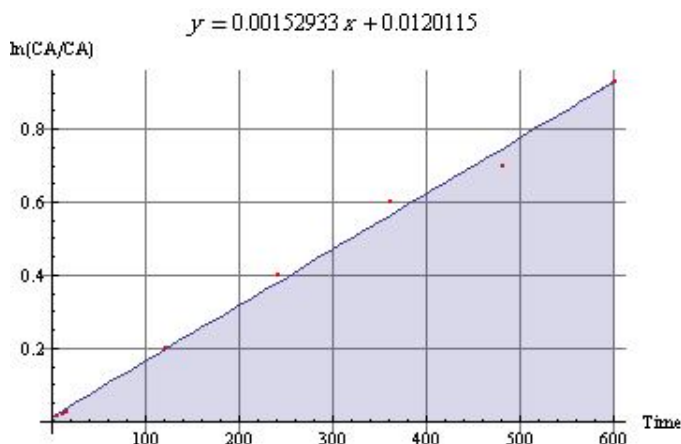


Figure 3: Representation of the hexamine dinitrate concentration-time data at 25°C - First Order, $R^2=0.997851$.

Similarly, The rate of conversion of hexamine dinitrate into DPT at 25°C was found relatively higher than that at 15°C . Depletion of about 60% of hexamine dinitrate was achieved after 600 minutes as shown in Table 2.

Table 2: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 25°C .

Time (min.)	0	5	10	15	120	240	360	480	600
Hexamine dinitrate (mg / ml)	0.597	0.586	0.585	0.581	0.488	0.400	0.327	0.296	0.235

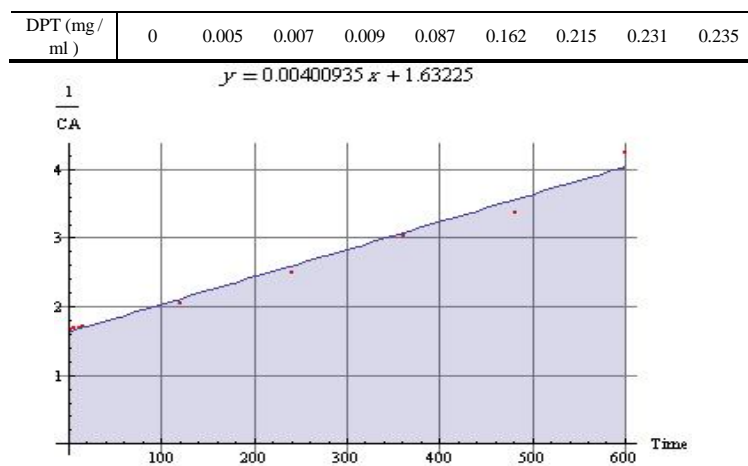


Figure 4: Representation of the hexamine dinitrate concentration-time data at 25°C - Second Order, $R^2=0.992881$.

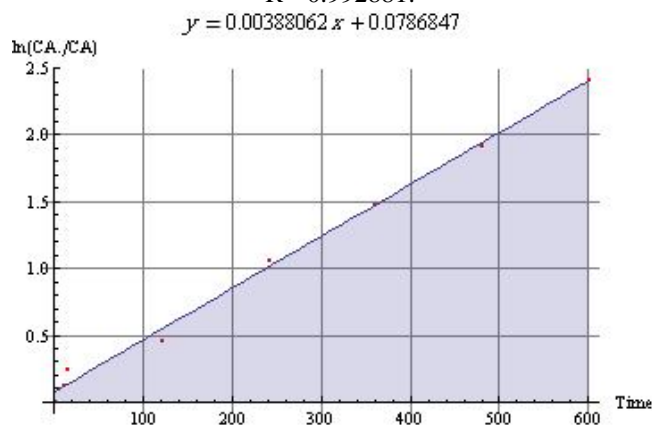


Figure 5: Representation of the hexamine dinitrate concentration-time data at 35°C - First Order, $R^2=0.99773$. The reaction rate was appreciably higher than that found below this temperature. About 90% conversion was recorded after 600 minutes.

Table 3: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 35 °C

Time (min.)	0	5	10	15	120	240	360	480	600
Hexamine dinitrate (mg / ml)	0.588	0.532	0.521	0.458	0.369	0.202	0.134	0.086	0.052
DPT (mg / ml)	0	0.034	0.050	0.102	0.178	0.315	0.368	0.401	0.438

Decomposition of hexamine dinitrate became faster than that found below this temperature. About two thirds of the hexamine dinitrate were depleted during the first fifteen minutes. This temperature has been already recommended by many authors

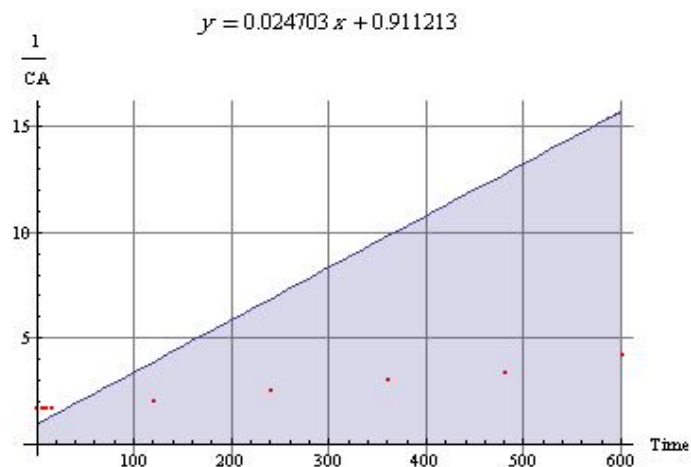


Figure 6: Representation of the hexamine dinitrate concentration-time data at 35 °C - Second Order, $R^2=0.953836$

Table 4: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 45 °C

Time (min.)	0	1	2	3	5	6	8	10	12	15
Hexamine dinitrate (mg / ml)	0.59	0.56	0.54	0.52	0.44	0.40	0.35	0.33	0.28	0.20
DPT (mg / ml)	0	0.02	0.03	0.05	0.12	0.14	0.19	0.20	0.25	0.32

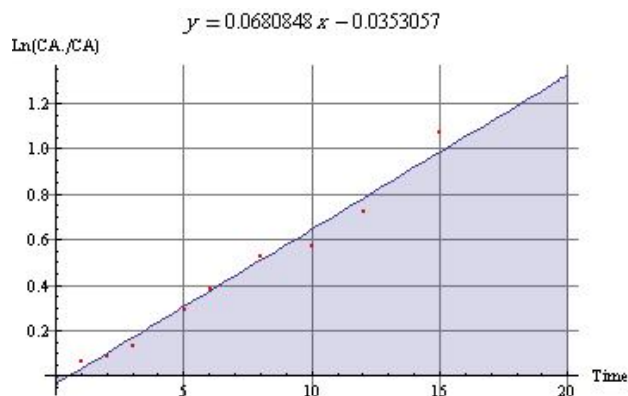


Figure 7: Representation of the hexamine dinitrate concentration-time data at 45°C - First Order, $R^2=0.990814$.

The concentrations and concentration terms found at 55 °C were neither fitting tightly to the first order nor to the second order ordinary kinetic models. The reaction at this temperature was faster. About 74% of the hexamine dinitrate were depleted during the first fifteen minutes. The formed DPT was somewhat less than the theoretical. This may be attributed to some sort of the side reactions. This

may also explain the recommendation given by many authors which limit the reaction temperature to 45 °C.

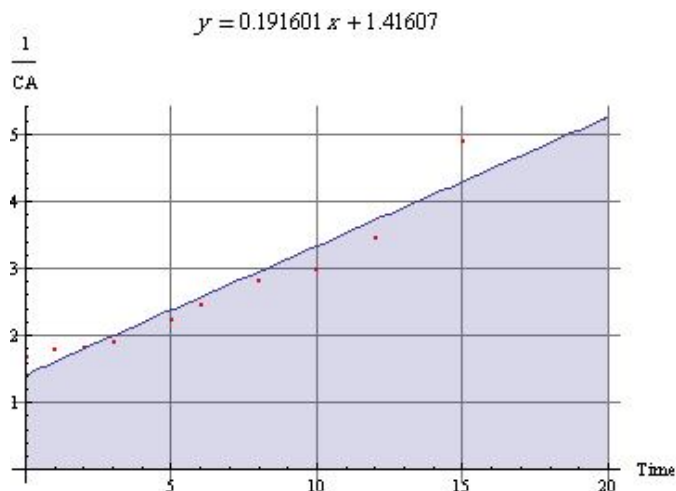


Figure 8: Representation of the hexamine dinitrate concentration-time data at 45 °C - Second Order, $R^2=0.960295$.

Table 5: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 55 °C

Time (min.)	0	5	10	15
Hexamine dinitrate (mg / ml)	0.5969	0.4082	0.3254	0.1552
	8	5	0	2
DPT (mg / ml)	0	0.1340	0.1875	0.2764
		0	0	0

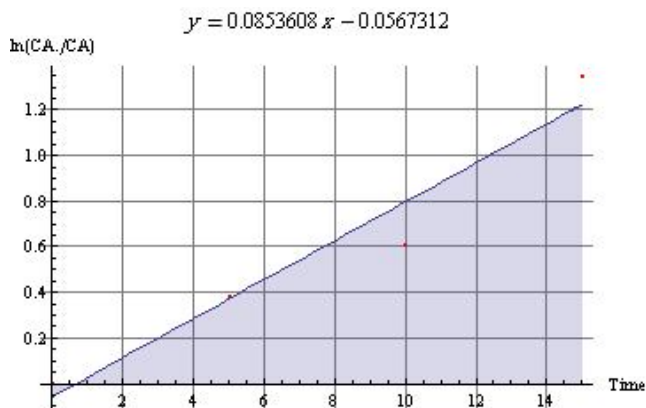


Figure 9: Representation of the hexamine dinitrate concentration-time data at 55 °C - First Order, $R^2=0.97128$.

The concentrations and concentration terms found at 65 °C were neither fitting tightly to the first order nor to the second order ordinary kinetic models. The reaction was as usual faster. About 77% of the hexamine dinitrate were

depleted in the first fifteen minutes. The DPT yield was less than that found in the literature.

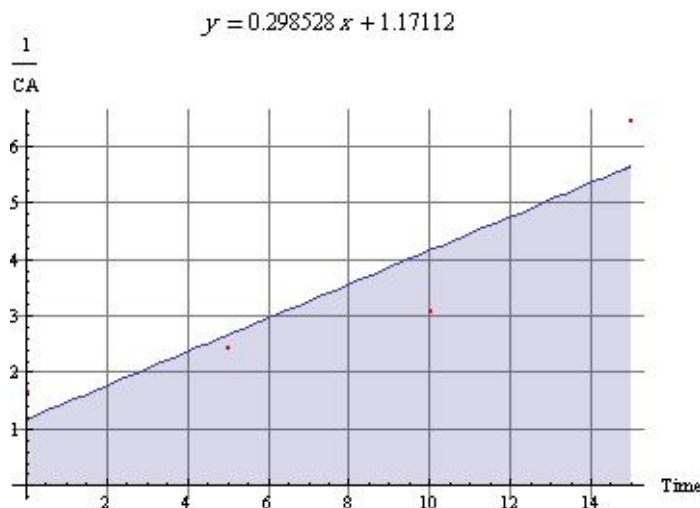


Figure 10: Representation of the hexamine dinitrate concentration-time data at 55 °C - Second Order, $R^2=0.917158$.

Table 6: DPT and hexamine dinitrate concentrations and concentration terms at different reaction times at 65 °C

Time (min.)	0	5	10	15
Hexamine dinitrate (mg / ml)	0.59780	0.39850	0.28370	0.14048
DPT (mg / ml)	0	0.14200	0.19230	0.26235

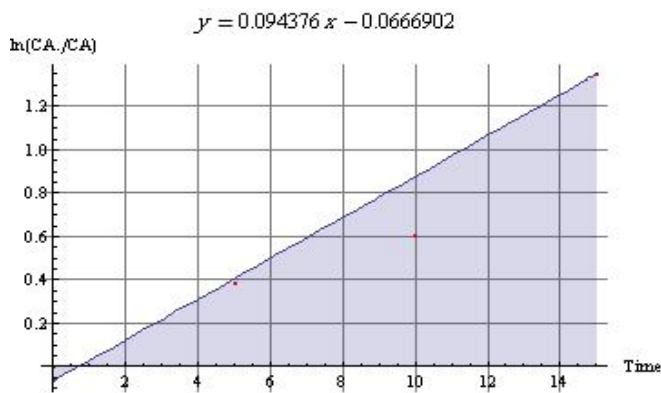


Figure 11: Representation of the hexamine dinitrate concentration-time data at 65 °C - First Order, $R^2=0.988583$.

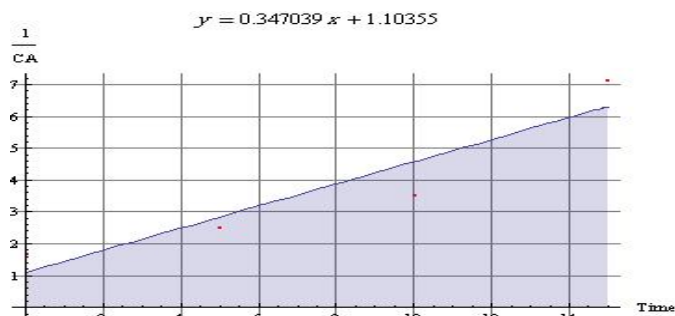
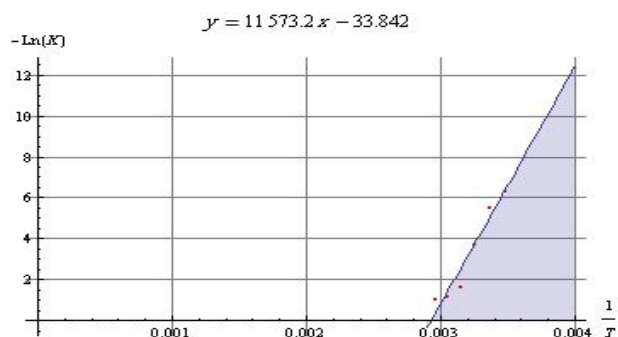


Figure 12: Representation of the hexamine dinitrate concentration-time data at 55 °C - Second Order, $R^2=0.934405$.

Table 7: Determined values of reaction rate constant of hexamine dinitrate conversion into DPT at different temperatures for the first and second order kinetic model.

Temperatur e (°C)	Rate Constant(k)		Correlation Factor(R^2)	
	1-st order(s^{-1})	2-nd order ($L.mol^{-1}.sec^{-1}$)	1-st order	2-nd order
15	0.0009	0.0018	0.9956	0.9883
25	0.0016	0.004	0.995	0.9858
35	0.0041	0.0247	0.9908	0.9098
45	0.0645	0.1915	0.9773	0.9222
55	0.0805	0.2985	0.9386	0.8412
65	0.0887	0.347	0.9658	0.8731



At each temperature, the analysis was done at different reaction times. The above mentioned figure shows the chromatogram obtained at the end of the investigated reaction time at 45°C. By examining the obtained chromatograms, the prepared compounds were identified and quantified. Concentrations of both unreacted and formed species were determined, where C_A and C_A are the initial and final concentrations of hexamine dinitrate respectively. The complete data and calculated results of HPLC analysis are summarized in previously mentioned Tables. As a first approximation, the reaction may be treated as a homogeneous one. From the concentration results, namely $\ln(CA/C_A)$ and $(1/C_A)$, shown above, the rate constant was calculated for first and second order kinetic models respectively as shown in the following Figures 13

and 14. The obtained values of rate constant are summarized in Table 7.

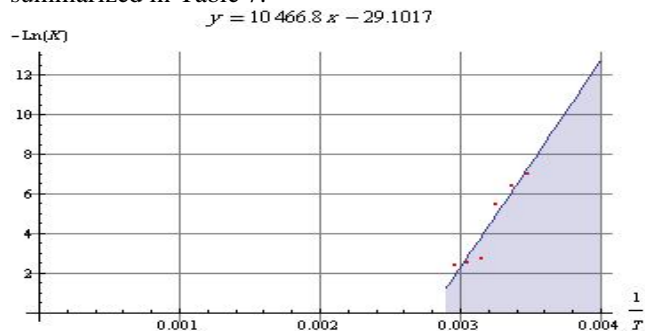


Figure 13: Representation of the hexamine dinitrate concentration – time data at 45 °C, according to first order kinetic models.

Similarly, Mathematica is used to obtain more accurate and optimized results. The arte constant is simply calculated from the obtained concentration results as shown in Figure 13-14 based on the calculated results from Table 7. [45]

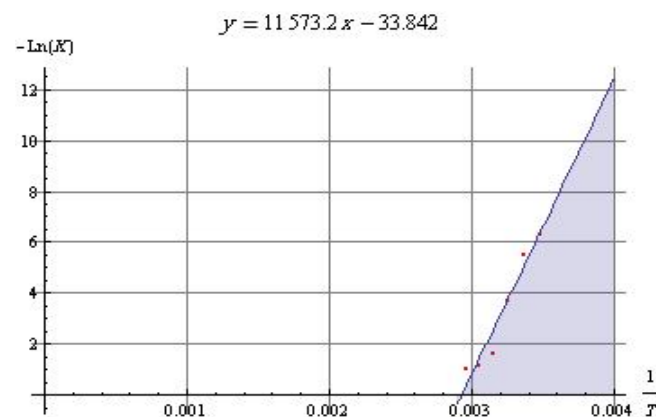


Figure 14: Representation of the hexamine dinitrate concentration – time data at 45 °C, according to second order kinetic models.

From Table 7, it is clear that the calculated rate constant, according to the first order model, is nearly doubled for each decade of temperature rise up to 35 °C. However, at 45°C the rate constant increases almost sixteen times.

Further increase of temperature, nearly 55°C and 65°C, has a slight effect on the rate constant. The results confirm the reported data in literature [11] that the optimal reaction temperature is 45°C. On the other hand, for the second order kinetic model, the increase of the rate constant with temperature is irregular and unjustifiable, especially at low temperatures. Moreover, the correlation factor values for the second order are inferior to those obtained for first order kinetic model. Arrhenius equation was used to calculate the activation energy of the reaction under investigation. The relation between $\ln(k)$ and $(1/T)$ for both first and second order kinetic model is shown in Figures 13-14.

The activation energy of the reaction was investigated using Arrhenius equation. The proposed kinetic model was found to be second order kinetic model based on the experimental data that were processed in order to find a relation between $\ln(k)$ and $(1/T)$ as shown in Figure 13-14.

The data in Table 7 are linearly interpolated with *Mathematica 10* to get a relation between the slopes k of the reactions with the temperature, and the data with the corresponding line are plotted in figure 13-14.

4. CONCLUSION

In conclusion, Mathematica was used as a powerful program for computing both numeric and algebraic calculations in many fields of science now such as physics, engineering, chemistry and even biology. Mathematica is one of the most powerful tools for dealing with data nowadays. It is used with any kind of data regardless of its nature whether biological, chemical or engineering data. Mathematica arrange and summarize the data. It also describe it by interpolating for best possible curves and then any kinds of graphs can be achieved. Also after the process of summarizing and interpolation. It is straight forward calculations to get the required statistical parameters of the data and also to measure the strength of interaction between different sets of data

We report here, optimizing the kinetic data DPT via using Mathematica. It was determined that the order of the reaction is a second-order reaction. The work reports the synthesis of DPT as an important and key intermediate for the synthesis of RDX and HMX. Recently, new research efforts have been devoted to increase the performance of energetic materials using nanocomposites and nanoparticles due to its unique properties not only in the field of energetic materials but also in other applications including medical, environmental, and industrial applications. [16-41]

The yield of the DPT prepared was about 65%; while the yield of the hexamine dinitrate prepared was about 89%. The analysis of the prepared samples using an efficient HPLC was a very reliable procedure. Decomposition of hexamine dinitrate became faster than that found below this temperature. About two thirds of the hexamine dinitrate were depleted during the first fifteen minutes. This temperature has been already recommended by many authors. The temperature of 45 ± 2 °C is the optimum temperature that gives the optimum conversion and yield. The analysis of the prepared samples using an efficient HPLC was a very reliable procedure. The concentrations and concentration terms found at 45°C were not fitting tightly to the ordinary second order kinetic model. Decomposition of hexamine dinitrate became faster and faster than that found below this temperature. About two thirds of the hexamine dinitrate were depleted during the first fifteen minutes. This temperature has been already recommended by many authors [6,10]. The applicability of the first order model which gave higher correlation coefficient when compared with the second.

ACKNOWLEDGEMENT

We express our deep gratitude to British University in Egypt (BUE). This work was partially performed using the facilities at the Nanotechnology Research Centre (NTRC) at the British University in Egypt (BUE). The authors express their deep gratitude for the financial support of the research project entitled “Synthesis and kinetic study of a selected energetic material” that has been provided by The Military Technical College, Cairo, Egypt.

The authors also thank Prof. M. M. Selet and Prof. Said M. A. Hassanein for their kind help, guidance, and support during all steps of this work, The School of Chemical Engineering at Military Technical College for using their facilities, and we also thank Prof. Shoukry M. Fayed, Prof. Omar Al-Farouk and Prof. Ahmed Baraka for their kind and valuable scientific comments and support which enabled us to finish this work.

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