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Highlights

- Drive Mobile Robot
- Control of 2-R Robot
- Pipeline Crawling Robot
- Fullerene and their Bromination

Discovering Thoughts, Inventing Future

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Kinematics, Localization and Control of Differential Drive Mobile Robot

By Sandeep Kumar Malu & Jharna Majumdar
Nitte Meenakshi Institute of Technology, India

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Keywords: kinematics, odometric localization, PID speed control, differential drive robot, lyapunov stability theory.

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Kinematics, Localization and Control of Differential Drive Mobile Robot

Sandeep Kumar Malu [α] & Jharna Majumdar [σ]

Abstract- The present work focuses on Kinematics, Localization and closed loop motion control of a differential drive mobile robot which is capable of navigating to a desired goal location in an obstacle free static indoor environment. Two trajectory planning approaches are made (i) the robot is rotated to eliminate orientation error and then translate to overcome distance error (ii) Both rotational and translational motion is given to the robot to overcome orientation and distance error simultaneously. Localization is estimated by integrating the robot movement in a fixed sampling frequency. The control law is based on kinematics model which provides updated reference speed to the high frequency PID control of DC motor. Stability of proposed control law is validated by Lyapunov Criterion. Both experimental and simulation results confirm the effectiveness of the achieved control algorithms and their efficient implementation on a two wheeled differential drive mobile robot using an 8-bit microcontroller.

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I. Introduction

Autonomous mobile robots have various applications in the field of industry, military and security environment. The problem of autonomous motion planning and control of wheeled mobile robots have attracted lot of research interest in the field of robotics. Consequently engineers working on design of mobile robots have proposed various drive mechanisms to drive such robots. However the most common way to build a mobile robot is to use two-wheel drive with differential steering and a free balancing wheel (castor). Controlling the two motors independently make such robots to have good manoeuvring and work well in indoor environment. Mobile robots with such drive systems are a typical example of non-holonomic mechanisms due to the perfect rolling constraints on a wheel motion (no longitudinal or lateral slipping).

An asymptotic stable controller using Backstepping method for posture tracking and its stability has been validated [1] [7]. An Adaptive Controller [2] to compensate errors can further improve its stability. A non-linear control design [6] using feedback linearization can provide better performance than conventional linearized controller. Many authors [2] [3] have proposed methods to reduce odometry error caused by kinematic imperfection. A different approach of localization using RFID technology [5] is efficient, fast and cheap.

A neural network [4] based reactive navigation algorithm for mobile robot in unstructured environment while avoiding obstacles is found to be optimized in computation.

In the present paper, kinematics model and localization using optical encoder coupled with the DC motor of a differential drive robot is presented. This model itself is used as a motion controller in a closed loop control scheme. In the absence of workspace obstacles, the basic motion tasks assigned to wheeled mobile robots may be reduced to moving between two robot postures and following a given trajectory.

The paper is organized as follows: In section II, basic equations of Kinematics and Motion Model of the robot are reported. The Localization in indoor environment using optical encoder coupled with the motor is described in brief. In section III, Control law is proposed and its stability analysis is carried out based on Lyapunov theory. In section IV, PID speed control of motor is presented. Section V includes some simulation results. Section VI highlights implementation strategies of control and localization using an 8-bit ATmega 32 microcontroller while Section VI contains conclusion and future work.

II. Kinematics of Differential Drive Robot

a) Motion Model

Let Inertial Reference Frame is $\{X_I, Y_I\}$ and Robot Frame is $\{X_R, Y_R\}$. The Robot position P [x y θ] is expressed in cartesian co-ordinate system of inertial frame. The relationship between Inertial and Robot frames is represented using basic transformation matrix as follows:

$$\dot{\varepsilon_R} = R(\theta)\dot{\epsilon_I}$$

$$= R(\theta).[\dot{x} \quad \dot{y} \quad \dot{\theta}]^T \qquad (1)$$

Where, $R(\theta) = \begin{bmatrix} \cos\theta & \sin\theta & 0 \\ -\sin\theta & \cos\theta & 0 \\ 0 & 0 & 1 \end{bmatrix}$

Author α: Research Associate in Robotics Lab at Nitte Meenakshi Institute of Technology, Bangalore, India.
e-mail: sandeepmalu77@gmail.com
Author σ: Prof and Head, Dept. of CSE (PG) at Nitte Meenakshi Institute of technology, Bangalore, India.
e-mail: jharna.majumdar@gmail.com

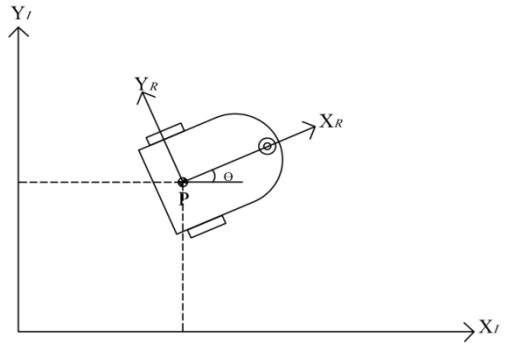

Fig. 1 : Representation of Robot on a Cartesian coordinate system

The robot under consideration is a two wheeled differential drive robot, where each wheel is driven independently. Forward motion is achieved when both wheels are driven at the same rate, turning right is achieved by driving the left wheel at a higher rate than the right wheel and vice-versa for turning left. This type of mobile robot can turn on the spot by driving one wheel forward and second wheel in opposite direction at the same rate. Third wheel is a castor wheel needed for the stability of mobile robot.

Each individual wheel contributes to the robot's motion and at the same time, imposes constraints on robot motion. It is assumed that the wheels of the robot do not slide. It is expressed by Non Holonomic Constraint.

$$\dot{x}\sin\theta - \dot{y}\cos\theta = 0 \qquad (2)$$

Also the measure of the traveled distance travelled by each wheel is not sufficient to calculate the final position of the robot. One needs to know how this movement is executed as a function of time. This can be illustrated in Fig. 2.

$$s_{1R} = s_{2R}\ ;\ s_{1L} = s_{2L}\ and\ S_1 = S_2$$
$$x_1 \neq x_2\ ;\ y_1 \neq y_2$$

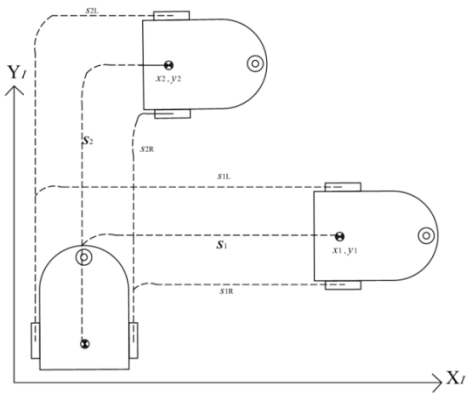

Fig. 2 : Dependence of Robot position on its velocities as function of time

The actual robot motion commands are the angular velocities w_R and w_L of the right and left wheel respectively, rather than robot driving and steering velocities v and ω. First consider the contribution of each wheel's spinning speed to the translation speed at *P* in the direction of $+X_R$. If one wheel spins ($v_1 = rw_R$) while the other wheel contributes nothing and is stationary ($v_2 = 0$), Since P is halfway between the two wheels, it will move instantaneously with half the speed i.e. $v_X = (1/2)rw_R$. In a differential drive robot, these two contributions can simply be added to calculate thev_X. Consider, for example, a differential robot in which each wheel spins with equal speed but in opposite directions. The result is a stationary, spinning robot. As expected, v_X will be zero in this case. The value of v_Y is even simpler to calculate. Neither wheel can contribute to sideways motion in the robot's reference frame, hence v_Y is always zero. Finally, we must compute the rotational component w of the robot. Once again the contributions of each wheel can be computed independently and just added. Consider the right wheel (we will call this wheel 1). Forward spin of this wheel results in counter clockwise rotation at point *P*. Recall that if wheel 1 spins alone, the robot pivots around wheel 2. The rotation velocity w_1 at P can be computed because the wheel is instantaneously moving along the arc of a circle of radius *d*. $w_1 = \frac{r}{d}w_R$. The same calculation applies to the left wheel, with the exception that forward spin results in clockwise-rotation at point *P*:

$$w_2 = -\frac{r}{d}w_L .$$

Mapping between Robot velocities to wheel velocities is given as follows:

$$v = v_X = v_1 + v_2 = r\left(\frac{w_R + w_L}{2}\right)\ ;v_Y = 0$$

$$\omega = w_1 + w_2 = r\left(\frac{w_R - w_L}{d}\right) \qquad (3)$$

Where, *r* = radius of wheel and *d* = axial distance between wheels.

b) Kinematic Equations

Kinematics is the most basic study of how mechanical systems behave. In mobile robotics, we need to understand the mechanical behaviour of the robot both in order to design appropriate mobile robots for desired tasks and to understand how to build control software.

Consider a differential drive robot at some arbitrary position *P* (q_c) [x_c, y_c, θ_c] which has a non-zero distance with the goal position *R* (q_d) [x_d, y_d, β] defined with respect to global inertial frame.

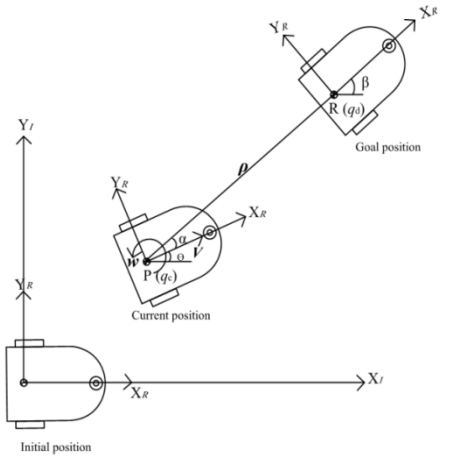

Fig. 3 : Error vectors of robot's position and orientation

The coordinate system of the robot is governed by combined action of both the linear velocity v and the angular velocity w. Using geometrical assumption the robot cartesian is given by:

$$\dot{x} = v\cos\theta$$
$$\dot{y} = v\sin\theta$$
$$\dot{\theta} = w \qquad (4)$$

Where $vcos\theta$ and $vsin\theta$ are the components of v along its X and Y axes and x, y and orientation θ are measured with respect to the reference Inertial Frame.

Same way position of the robot can also represented in terms of polar coordinates involving error distance ρ > 0 as:

$$\dot{\rho} = -vcos(\beta - \theta) = -vcos\alpha$$
$$\dot{\beta} = v\frac{sin\alpha}{\rho}$$
$$\dot{\theta} = w$$

Now, $\alpha = \beta - \theta$ be the angle measured between robot axes frame [X_R, Y_R] and the distance vector frame ρ also $\rho = \sqrt{(x_d - x_c)^2 + (y_d - y_c)^2}$ as per distance formula between two coordinates system. Finally we conclude

$$\dot{\rho} = -vcos\alpha$$
$$\dot{\alpha} = -w + \frac{vsin\alpha}{\rho}$$
$$\dot{\beta} = \frac{vsin\alpha}{\rho} \qquad (5)$$

Kinematics equation based on polar coordinates (5) is valid when $\rho \neq 0$ and above set of equations will be employed in establishing feedback control law for robot manoeuvring as discussed later in section III.

c) Odometric Localization

Localization is one of the most fundamental aspects of a mobile robot. All mobile robot system has to answer the fundamental question, which is "Where Am I", i.e. the current location & orientation of the robot has to be obtained, so that the robot can easily move from source to destination. There are number of localization techniques with respect to mobile robot, however in the present work we have used dead reckoning method for localization. Dead reckoning method uses odometry to measure the movement of the robot. In the present work, we obtain the data from an incremental encoder (odometry), which is fitted along with a motor of the mobile robot. Incremental encoders measure the rotation of the wheels, which in turn, calculates robot position and orientation using integration approaches of kinematic model over [t_k, t_{k+1}].

Assuming Robot configuration q_k $[x_k \quad y_k \quad \theta_k]$ and constant velocity inputs V_k and w_k are known at discrete time t_k, then using Euler integration

$$x_{k+1} = x_k + v_k T_s \cos\theta_k$$
$$y_{k+1} = y_k + v_k T_s \cos\theta_k$$
$$\theta_k = \theta_k + w_k T_s \qquad (6)$$

where, $v_k T_s = \Delta s$ and $w_k T_s = \Delta\theta$
$T_s = t_{k+1} - t_k$

The reconstruction of the current robot configuration is based on the incremental encoder data (odometry). Let $\Delta\varphi_R$ and $\Delta\varphi_L$ be the no. of wheel rotations measured during the sampling time T_s by the encoders. Linear and angular displacements of the robot is given as

$$\Delta s = \frac{r}{2}(\Delta\varphi_R + \Delta\varphi_L)\ ,\ \Delta\theta = \frac{r}{d}(\Delta\varphi_R - \Delta\varphi_L) \qquad (7)$$

Where, r = radius of wheel and d = axial distance between wheels.

For a differential-drive robot the position can be estimated starting from a known position by integrating the movement (summing the increment travel distances). The estimate of robot configuration at time t_k is computed as:

$$\begin{bmatrix} x_k \\ y_k \\ \theta_k \end{bmatrix} = \begin{bmatrix} x_{k-1} \\ y_{k-1} \\ \theta_{k-1} \end{bmatrix} + \begin{bmatrix} \cos\theta & 0 \\ \sin\theta & 0 \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \Delta s \\ \Delta\theta \end{bmatrix}, \qquad (8)$$

Robot localization using the above odometric prediction (commonly referred to as *dead reckoning*) is accurate enough in the absence of wheel slippage and backlash.

III. Control Law and Stability

The control algorithm must now be designed to drive the robot from its current configuration; say (x_c, y_c, θ) to the goal position (x_g, y_g, β). Here the objective is to find a control $u = [v\ w]$ so that the robot's goal position is reached in finite interval of time. The proposed control law is state dependent i.e. $[v\ w] = f(\rho, \alpha, \beta)$ which guarantees the state to be asymptotically driven to [0, 0, β] without attaining $\rho = 0$

in finite time. One of the most commonly used methods to study the asymptotic behaviour is based on the Lyapunov stability theory. Consider a simple positive definite quadratic form of Lyapunov function:

$$V = V_1 + V_2$$
$$= \frac{1}{2}\rho^2 + \frac{1}{2}\alpha^2 \quad (9)$$

Where the parameters V_1, V_2 represent one half of the squared weighted norms of the "distance error vector" ρ and "orientation error vector" α exhibited by the robot between its current position and goal position defined with respect to the Reference Inertial Frame. Its time derivative is given by:

$$\dot{V} = \dot{V}_1 + \dot{V}_2 = \dot{\rho}\rho + \alpha\dot{\alpha}$$

Using kinematics equation (5),

$$\dot{V} = \rho(-v\cos\alpha) + \alpha(-w + \frac{v}{\rho}\sin\alpha) \quad (10)$$

From the equation (10), the first term can be made non-positive by letting the linear velocity of the form:

$$v = K_\rho \rho\cos\alpha \qquad K_\rho > 0 \quad (11)$$

then,

$$\dot{V}_1 = \rho(-K_\rho \rho cos^2\alpha)$$
$$= -K_\rho \rho^2 cos^2\alpha \leq 0 \quad (12)$$

This means that $\dot{V}_1$ term is always non-increasing in time and consequently, since it is asymptotically converges to non-negative finite limit.

Similarly the second term $\dot{V}_2$ can be made be non-positive by letting the angular velocity w take the form of:

$$w = K_\rho \sin\alpha\cos\alpha + K_\alpha\alpha \qquad K_\alpha > 0 \quad (13)$$

Then,

$$\dot{V}_2 = \alpha(-K_\rho \sin\alpha\cos\alpha - K_\alpha\alpha + \frac{K_\rho\rho\sin\alpha\cos\alpha}{\rho})$$
$$= -K_\alpha\alpha^2 \leq 0 \quad (14)$$

Finally leading to the following expression for the time derivative of the Lyapunov function V

$$\dot{V} = \dot{V}_1 + \dot{V}_2 = -K_\rho \rho^2 cos^2\alpha - K_\alpha\alpha^2 \leq 0 \quad (15)$$

The result in (15) is a negative semi-definite form. By applying Barbalat's Lemma, it follows that $\dot{V}$ necessary converges to zero for increasing time; thus in turn implying convergence of the state vector $[\rho, \alpha, \beta]$ to $[0, 0, \beta]$. Hence it can be concluded that control vectors expressed by (11) and (13):

$$v = K_\rho \rho\cos\alpha \ \ and \ w = K_\rho \sin\alpha\cos\alpha + K_\alpha\alpha\,,$$

makes the robot motion behaviour to be smooth and stable.

The overall feedback control architecture for Go-to-Goal motion can be summarized in Fig. 4.

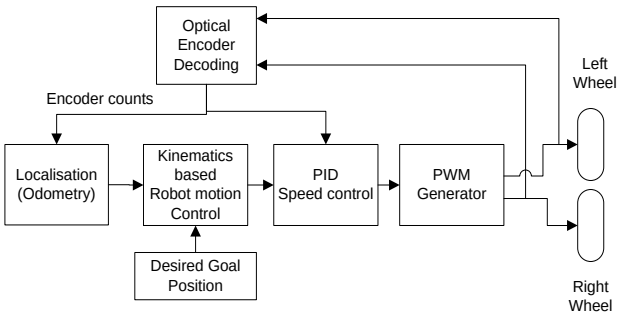

Fig. 4 : Block diagram of proposed Kinematics based motion controller

IV. Low Level pid Speed Control

The speed of DC motor can be adjusted to a great extent so as to provide easy control and high performance. There are several conventional and numeric controller types intended for controlling the DC motor speed. Recently, many modern control methodologies such as nonlinear control [8], optimal control [9], variable structure control [10] and adaptive control [11] have been extensively proposed for DC motors. However, these approaches are either complex in theory or difficult to implement. PID controller algorithm involves three parameters denoted *P*, *I* and *D* interpreted in terms of time. *P* depends on present error, *I* on the accumulation of past errors, and *D* is a prediction of future errors. PID control with its three term functionality covering both transient and steady-states response, offers the simplest and yet most efficient solution for many real world control problems. In spite of the simple structure and robustness of this method, optimally tuning gains of PID controllers have been quite difficult.

The electric equivalent circuit of the armature and the free–body diagram of the rotor are shown in the Fig. 5 below

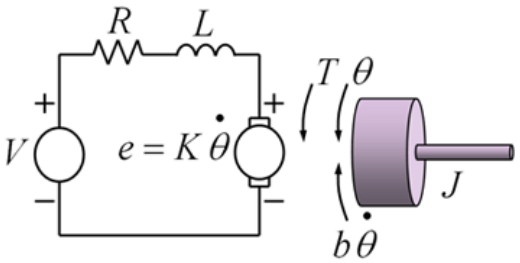

Fig. 5 : Circuit Diagram of DC motor

The dynamic equations and the open loop transfer function of the DC motors are:

$$s(Js + b)\theta(s) = KI(s)$$
$$(Ls + R)I(s) = V(s) - Ks\theta(s)$$
$$P(s) = \frac{s\theta(s)}{V(s)}$$
$$P(s) = \frac{K}{(Js+b)(Ls+R)+K^2}\left[\frac{rad/sec}{V}\right] \quad (16)$$

where,

J = moment of inertia of the rotor

b = motor viscous friction constant

K_e = electromotive force constant

K_t = motor torque constant

R = electric resistance

L = electric inductance

V = voltage across motor terminals

I = current flowing in the circuit

Here, $K_t = K_e = K$.

And the system schematic looks like:

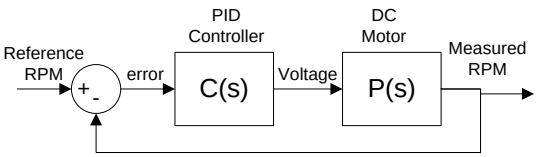

Fig. 6 : PID for DC motor speed control

The PID control design criteria are (i) less settling time (<1 s) (ii) overshoot less than 5% (iii) steady state error less than 1%. The PID speed control design is incorporated into the system. The transfer function for a PID controller given is by:

$$C(s) = K_P + \frac{K_I}{s} + K_D s \qquad (17)$$

Where, K_P, K_I and K_D are gains. PID is simulated in Matlab and its response is plotted. Further, gain values are tuned manually to obtain desired response.

V. Simulation Results

Simulation results of DC motor characteristics incorporating PID for unit step input at different gains are shown in figure 7:

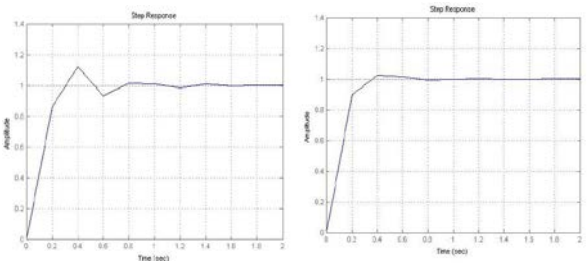

Fig. 7 : Unit step response at a) K_P=0.2, K_I=39, K_D =0.25; b) K_P=5, K_I=8, K_D =1.5

Simulation results are performed to illustrate the effectiveness of the proposed control law. Here sets of five position coordinates [0, 0, 0], [5, 0, 90˚], [5, 5, 180˚], [0, 5, -90˚] and [0, 0, 0] are considered to traverse a square path. Both the approaches of reaching the goal position are depicted below.

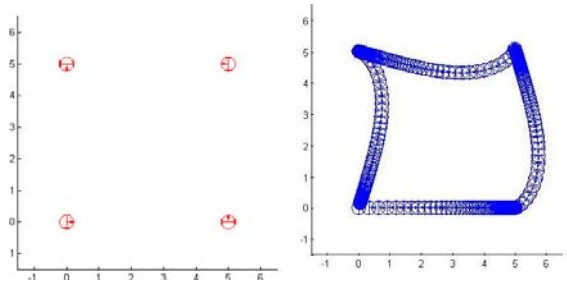

Fig. 8 : Posture tracking with simultaneous translation and rotation motion

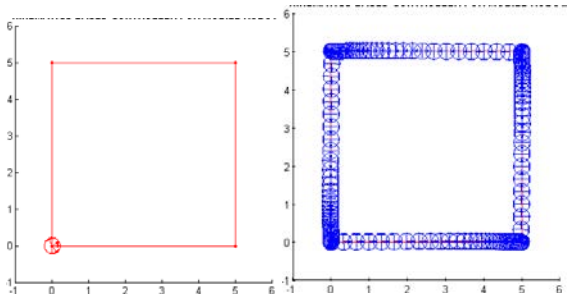

Fig. 9 : Posture tracking with first rotate then translate motion

From the plots, it can demonstrated that the proposed control law show good convergence in both the cases and is equally applicable in all conditions of robot movement between initial and goal position.

VI. Implementation

The robot which we used for our experiments is fabricated in-house. It has a differential-drive mechanism consisting of high torque Dynaflux DC motors each of which is equipped with high resolution Jayashree-15S optical quadrature shaft encoder for precise position and speed feedback to controller. The robot uses two 12V DC batteries in series to power motors, while 5V for Microcontroller development board. The power electronics module used to drive the DC motor is Super Hercules 9V-24V, 15A Motor Driver from Nex-robotics. The algorithm of localization and control has been implemented on 8-bit Atmega32 Microcontroller.

The output signal from one channel of encoder is fed to the rising edge enabled external interrupt pin. On interrupt, the status of other channel decides the direction and equivalent rotation counts of robot motion. These counts on accumulation at fixed sampling frequency (in our case 33 Hz) resolve robot position estimation using the expression for odometric localization explained in section II. Also these counts acts as feedback to PID speed control executing at 100 Hz frequency.

The control algorithm runs at 33 Hz wherein distance error vector and orientation error vector between robot instantaneous position and goal (target) position are calculated and robot control vectors viz.

linear velocity v and angular velocity w are obtained. Two approaches to reach the goal position have been implemented. In first one, robot is made to rotate until orientation error is eliminated and then translated to overcome distance error. While in other method, robot exhibits both linear and angular velocities to overcome distance and orientation error simultaneously.

Using Kinematics motion models the linear and angular velocities of the robot are transformed to right and left wheel speeds and fed as reference speed to PID speed control. The upper limit of robot motion is set to 40 RPM.

Fig. 10 : Experimental Differential Drive Robot

VII. Conclusion and Future Work

In this paper, Kinematics based feedback path controller for a differential drive mobile robot has been presented. Both the approach of Go-to-Goal motion is implemented using 8-bit Atmega 32 microcontroller. The robot motion behaviour in real time is in line with the behaviour observed in simulation. Also the proposed control law is validated as per Lyapunov stability criterion. The control algorithm proposed in this paper will be extended for human following application, where set points (goal locations) are updated regularly through computer vision algorithm of detecting and tracking human.

The present work will be extended to include obstacle avoidance in Go-to-Goal motion and dynamics consideration of robot for control which can enhance better stabilization. Further, Umbmark calibration needs to be performed to avoid localization error due to irregular wheel diameter and wheel axial alignation. Also Attitude compensation has to be incorporated in odometric localization whenever robot travels in irregular surface.

VIII. Acknowledgement

The authors express their sincere gratitude to Prof. N R Shetty, Director and Dr. H C Nagaraj, Principal for their encouragement and support to undertake multidisciplinary research at NMIT. The authors thank the Mechanical Engineering team of NMIT led by Prof. Sunil Kumar H S for designing and fabricating the robot. The authors also wish to acknowledge Mr R Praveen Jain and Mr Srikanth A Rao, former Research Associates, Robotics Lab NMIT for their contribution in the work.

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Thermal Stability of Foils Made of Graphene-Oxide and Grapheme -Oxide with Fullerene and their Composites with Methyl Car Boxy Cell u lose and with Beta 1, 3/1, 6 – D-Glucan

By Klouda Karel, Zemanová Eva, Friedrichová Romana, Brádka Stanislav & Gembalová Lucie

VŠB-Technical University of Ostrava, Czech Republic

Abstract- This contribution contains data on thermal stability of certain materials whose initial precursor is graphite. Graphite was oxidized separately and in a mixture with fullerene C_{60}. The prepared oxides were processed with vacuum filtration to produce foils and their morphology and thermal stability was described. The graphene oxides reacted with nano-cellulose and ß- glucan to produce composites. The prepared composites in the form of foils were tested for thermal stability and further analyzed e.g. by FT-IR, SEM, etc.

Keywords: grapheme oxide, fullerene - c_{60} Intercalate, composite, nano-cellulose, ß-glucan.

GJRE-H Classification : FOR Code: 091099

Strictly as per the compliance and regulations of:

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Klouda Karel [α], Zemanová Eva [σ], Friedrichová Romana [ρ], Brádka Stanislav [ω] & Gembalová Lucie[¥]

Abstract- **This contribution contains data on thermal stability of certain materials whose initial precursor is graphite. Graphite was oxidized separately and in a mixture with fullerene C_{60}. The prepared oxides were processed with vacuum filtration to produce foils and their morphology and thermal stability was described. The graphene oxides reacted with nano-cellulose and ß- glucan to produce composites. The prepared composites in the form of foils were tested for thermal stability and further analyzed e.g.by FT-IR, SEM, etc.**

Keywords: grapheme oxide, fullerene - c_{60} Intercalate, composite, nano-cellulose, ß-glucan.

I. Introduction

Graphite is an allotropic modification of carbon with sp^2 bonds and made up of layers of mutually interconnected hexagonal rings. The layers are arranged in parallel planes 335 pm apart. Carbon atoms in the adjoining layers are not chemically bonded to each other and they are attached by weak van der Waals forces that make it possible for various atoms or molecules in liquid or gaseous form to get in between the carbon layers. The resulting substances are called intercalation compounds of graphite and their characteristic parameter is the so-called "degree of intercalation", which indicates the number of carbon layers between two layers of an inter calated substance (Klouda, 1985).

Depending on a type of the intercalated substance the graphite plane may be either an acceptor or donor of electrons. Another option is the so-called π–complex created by intercalation of substances of AX_y type, where A is a metal or non-metal with a high valence status, X is an electronegative element and y is a stoichio metric coefficient.

Author α: VŠB-Technical University of Ostrava, Faculty of Safety Engineering. State Office for Nuclear Safety, Prague.
e-mail: Karel.Klouda@sujb.cz
Author σ: State Office for Nuclear Safety, Prague.
e-mail: eva.zemanova@sujb.cz
Author ρ: Ministry of the Interior– General Directorate of the Fire Rescue Service of the Czech Republic, Technical Institute of Fire Protection.
Author ω: National Institute for Nuclear, Chemical and Biological Protection Kamenná.
Author ¥: VŠB-Technical University of Ostrava, Faculty of Mining and Geology.

Intercalates of graphite with alkali metals have been known since 1930s. They are called intercalates of the first degree with the formula C_8M (M=K, Rb, Cs), i.e. they are characterized by a stacking sequence of layers of carbon and alkali metal.

Intercalates of graphite with alkali metals or in combination with other metals have been used in a number of applications as catalysts, e.g. for synthesis of ammonia, synthesis of carbohydrates by hydrogenation of carbon oxides, hydrogenation of olefins, they have sorption properties etc. (Klouda, 1985). Substituents can be chemically bonded to graphite under certain conditions by fluorination or oxidization.

Fluorination of graphite with elemental fluorine at 400-600°C produces a covalent compound called fluoro graphite CF_x, x=0.25-1.12, depending on reaction conditions of the fluorination (Klouda, 1985). Oxidization of graphite with strong oxidizing agents produces grapheme oxide (GO), which is a precursor for chemical preparation of graphene (Makharza et. al., 2013).

Publications dealing with oxidization of graphite to prepare G-O usually specifya method use das described by specific authors: Hoffmann (HNO_3, $KClO_3$), Stay dennaier (HNO_3, $KClO_3$), Tour (P_2O_5, $KMnO_4$), Hummers ($NaNO_3$, $KMnO_4$). In all those methods the mainchemical agent used is concentrated sulfuric acid (Chang and Pumera, 2013).

GO is a compound made up of a carbon skeleton with main functional groups, such as carboxyl, carbonyl, epoxy and ether group sandhydroxy-groups. These functional group senable chemical lreactions of GO (Zang et. al., 2011) to form covalent bonds with other compounds (e.g.esterification, amidation).

Another option is a GO reaction to form non-covalent bonds (Makharza et. al., 2013). The possible types of the bonds are hydrogen bonds, van der Waals forces, H-π, cation -π, anion-π, π–π, electrostatic forces. These non-covalent bonds are employed in preparation of composite polymers, biopolymers (Yoo, B. M. et. al., 2013) and in use of GO adsorption and absorption properties (Kyzas et. al., 2014; Fakhri et al., 2013; Chabot et. al., 2014).GO suspension can be vacuum filtered to prepare foils that find use in biology, electrical

engineering, optics (Russo et. al., 2013) and biomedicine (She n et. al., 2012).

Graphene can be prepared by a chemical method which consists in reduction of oxidized carbon (functional groups) in GO with various reducing agents (hydrazine , metal hydride, hydrogen, hydrogen iodide) or reducing methods, such as reflux in a polar solvent, microwaves irradiation, electrochemical reduction (Dreyer et. al., 2010).

The composition of graphene-oxide and its decomposition by an exothermic creaction in dicates a potential fire risk. This shall apply mainly to industrial production, processing and storage of GO-materials. If stored in a solid form GO shall be protected against sources of heat, electric discharges and exposure to high-intensity light. A question which appeared in publications by Krishnan et al. (Krishnan et al., 2012), i.e. whether GO is afire retarder or fire hazardous material, has defined the objective of this work in which we want to assess behavior of GO and GO-C_{60} foils and their composites with cellulose and ß-glucan when thermally exposed.

a) Experimental Part

i. *Employed chemicals*

Graphite PM – very fine cry stall in epowder graphite, mesh0.025mm, Supplier: Koh-I-Noor Netolice, Czech Republic

Fullerene C_{60}, 99.5%purity, SES Research, Houston USA

Sulfuric acid, nitric acid, potassium permanganate, sodium hydro carbonate, CM –cellulose C4146 – Supplier: Sigma –Aldrich

Beta 1, 3/1, 6 – D – glucan (59% beta, 9% alpha), botanic source oyster mushroom, Supplier: Dimenzia s.r.o., Kežmarok Slovakia.

ii. *Employed methods*

Ultrasonification with PS4000A, power output 500W, thermostat 0-77°C, frequency 35 kHz

ATR analysis by means of FTIR spectrometry was performed using the spectrometer Brucker Aplha/FT-IR, ART crystal (identified as Platinum Diamond 1 Ref1), software OPUS 6, 5, source IR SiC Globar. The number of spectrum scans was 24, resolution 4 cm^{-1}, spectrumrange 375-4000 cm^{-1}.

Thermal analyses TGA and DSC of the prepared an of ibers were performed on STA 1500, Instrument Specialists Incorporated-THASS, analytical scale SUMMIT, SI 234-4, at flowrate 20 ml/min., heatingrate 10°C/min., ceramiccrucible, diameter 5 mm and height 8 mm, degradation medium: air.

Morphology of then an of ibers was determined with SEM Phenom FEI and SEM FEI Quanta 650 FEG (USA).

b) Preparation of Graphene-Oxide (GO) and Graphene-Oxide + C_{60}(GO-C_{60})

Graphite was oxidized with a mixture of H_2SO_4, $KMnO_4$ and $NaNO_3$ according to Hummers and Offer man (Hummers and Offer man, 1958). Graphite, sulfuric acid and sodium nitrate (for experiments I-II also fullerene C_{60}), were placed into a flask, the mixture was stirred and cooled to 10°C. Potassium permanganate was subsequently added into the reaction mixture through a hopper in small doses. The mixture with the permanganate was slowly heated to 60°C and stirred at that emperature for 3 hours. Then it was left to stand for three days at the laboratory temperature.

The obtained product was filtered off, washed with big quantity of distilled water until negative reaction to sulfate anions and dried for three days on a Petridish at 50-60°Ctoform foils of GO or GO-C_{60}.

Samples weights for the individual experiments I –III

- I and II: 1 g graphite PM; 35 ml H_2SO_4; 2.11 g $NaNO_3$; 0.5 g C_{60} and 4.6 g $KMnO_4$
- III.: 2 g graphite PM; 45 ml H_2SO_4; 2.8 g $NaNO_3$ and6.5 g $KMnO_4$

c) Oxidation of Fullerene Alone C_{60} (Blank Test)

In order to confirm or to refute our the oretical assumptions about behavior of fullerene in an oxidation mixture we have performed an experiment in which we maintained mutual ratios of carbon to the other reagents as those used in the experiment with graphite (0.7g C_{60}, 1 g $NaNO_3$, 2 g $KMnO_4$, 18 ml H_2SO_4). Also there action times and subsequent treatment were equivalent. After vacuum filtration we did not obtain foils but after the drying we obtained black loose powder (hereinafterC_{60}-oxi). The powder was investigated with FT-IR, TGA, DSC analysis and the results were compared with analyses of the initial fullerene.

d) Reaction of GO and GO-C_{60} Foils with Cellulose in Acid Environment

GO (0.3 g) and GO-C_{60} (0.3 g) foils were placed into Erlenmeyer flasks and 10 ml of distilled water was added. Foil schanged into suspension safter 3 day so firregularstirring and short-termultrasonification. Subsequently, cellulose was added into the flasks (0.65 g) and 8 ml H_2SO_4 (96%). The reaction was exothermic. The mixture was ultrasonificated in a water bath at 40°C. Then the flask content was poured into 50 ml of distilled water and neutralized with a solution of sodium hydro carbonate ($NaHCO_3$) until neutral reaction. The product was then vacuum filtered and washed on a filter with ca. 40 ml of distilled water, dried at 50°C on a Petri dish on which it formed foils.

e) Reaction of GO with ß-Glucan

Graphene oxide (prepared according to Hummers, 1958) in the form off oils (0.3g) was placed

into Erlenmeyer flasks with 25 ml of distilled water. No suspension was formed after 48 hours and it was necessary to performer peated ultrasonification 5x 2 minutes to prepare the suspension. Beta – glucan (0.45 g) was added into the suspensions and in one of the flasks also 0.7 ml of concentrated H_2SO_4 (an exothermic process occurred after the acid was added).

i. *Visualization of the flask content*

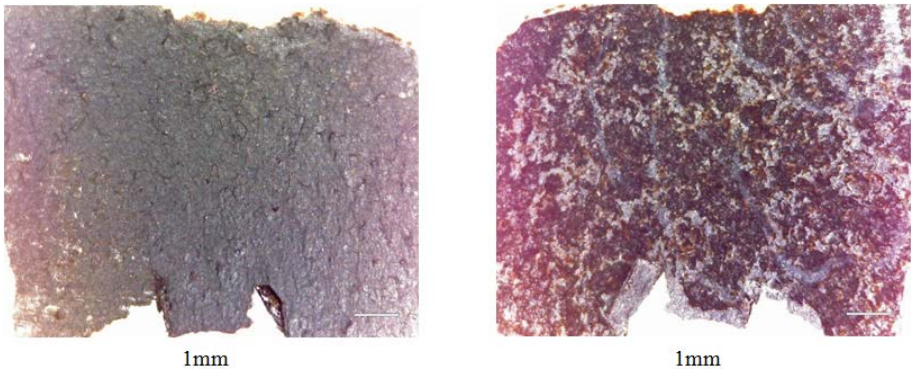
- without H_2SO_4 gel (product I)
- with H_2SO_4 suspe nsion(product II)

Subsequently, Erlenmeyer flasks were placed into a water bath (40°C) and ultrasonificated 3 times for 2 minutes. Then they were left to stand for 15 day sat the laboratory temperature. The flask with the product I (GO-ßG) was vacuum filtered and the resulting black foil was dried at 50-55°C. The flask with the product II (GO-ß G, H+) was poured into a 300 ml flask, diluted with 200 ml of distilled H2O and subsequently 12 times decanted top H 6.5. The flask contained brown spongycoagu late in1/3 of the flask volume. A part of the coagulate was vacuum filtered and it formed a brown foil which was dried at 50-55°C.

II. Results

a) Oxidation of Graphite and Mixture of Graphite-C_{60}

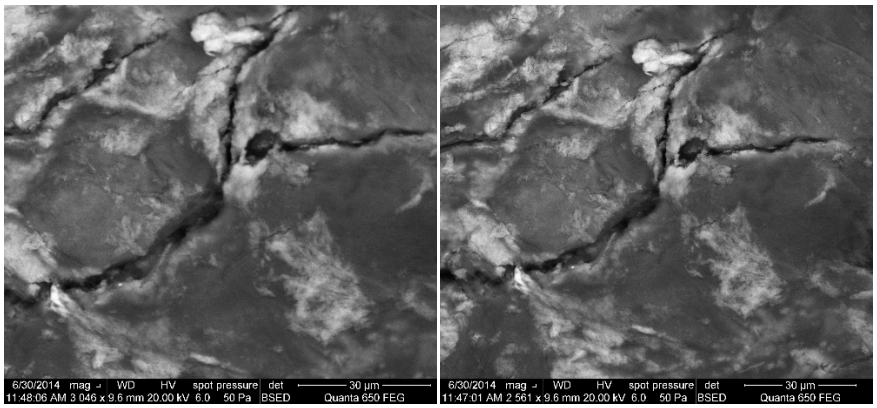
The foil sobtained by vacuum filtration from the product of oxidation of graphite alone and the product of joint oxidation with C_{60} had clearly different morphologies (see Fig. 1). GO-C_{60} foils looked more compact than GO foils. When inspecting the morphology with electron micros copy the GO-C_{60} foil has a rougher surface (see Fig.2). For GO-C_{60} foils we also determined its texture.



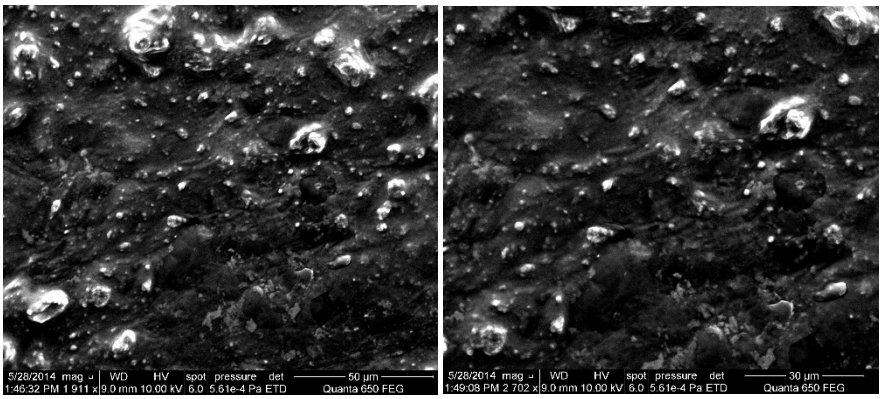
a) foil obtained by oxidation of graphite: front and back side of the foil, scale 1 mm

b) foil obtained by oxidation of graphite with fullerene C_{60}: front and back side of the foil, scale 1 mm

Figure 1 : Roughmorphology of the foils after vacuum filtration of GO (a) and GO-C_{60} (b)



a) GO: different views



b) GO-C_{60} : different views

Figure 2 : Fine morphology of the foils after vacuum filtration of GO (a) and GO-C_{60} (b)

The specific surface of the foil samples GO-C_{60} was 21.9m^2/g and the volume of adsorbed monomolecular layer was 5.03 ml/g. The volume of me so pores or macro pores was 0.286 ml/gin comparison with the volume of micro pores which was0.001 ml/g. The volume representation of me so pores was ca. 286 times higher than that of micro pores. The sample had a me spoor us character with some representation of macro pores. The sample contained very little micro pores (see the volume representation – only0.001 ml/g)consisting of the pores with the diameter 0-1 nm (ca. 63%) – only one fraction of the pores, while no other micro pore fractions were identified -they were probably clogged. Theme sopores included the following fractions: 1.5-3 nm (ca. 38%), 3-5 nm (ca. 13%) and 5-10 nm (ca.5%) and 10-50 nm (ca.8%). As for macro pores, the sample contained only one fraction, while ca. 24% of the specific surface was formed by macro pores with the diameter 50-200 nm. The measured parameters are shown in Table No. 1.

Table 1 : Texture parameters of GO–C_{60} samples

Sample identification	a[m^2/g]	B[cm^3/g]	c[cm^3/g]	d[cm^3/g]	e[cm^3/g]
GO-C_{60}	21.9	5.03	0.040	0.001	0.286

a – specific surface, b – volume of adsorbed monomolecular layer, c –cumul ative volume of pores, d –cumul ative volume of micro pores, e –cumulative volume of me so- and macro pores

Subsequently, the foils were examined with X-ray analysis (Fig.3), FT-IR, TGA and DSC analyses. The X-ray analysis has proved a negligible difference in expansion of the space between the layers, see Fig.3.

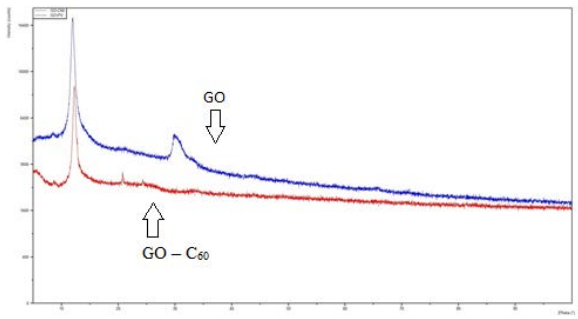
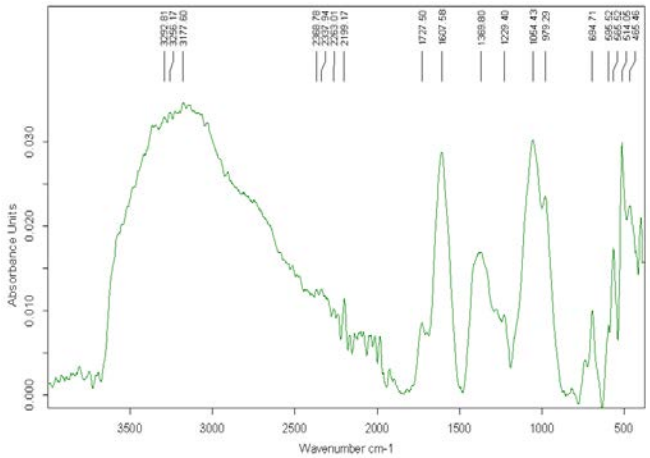
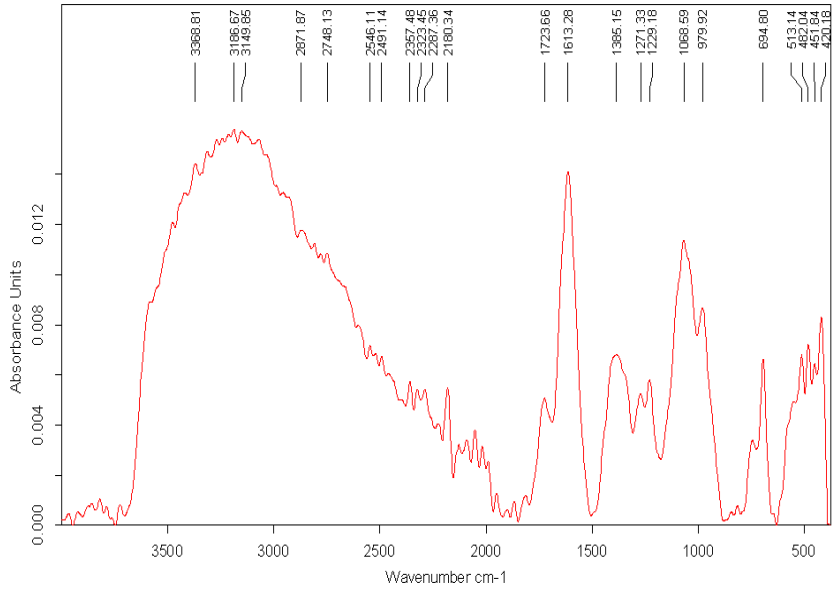


Figure 3 : X-ray analysis, GO: d=739 pm, 299 pm; GO-C_{60}:d= 718 pm, 425 pm

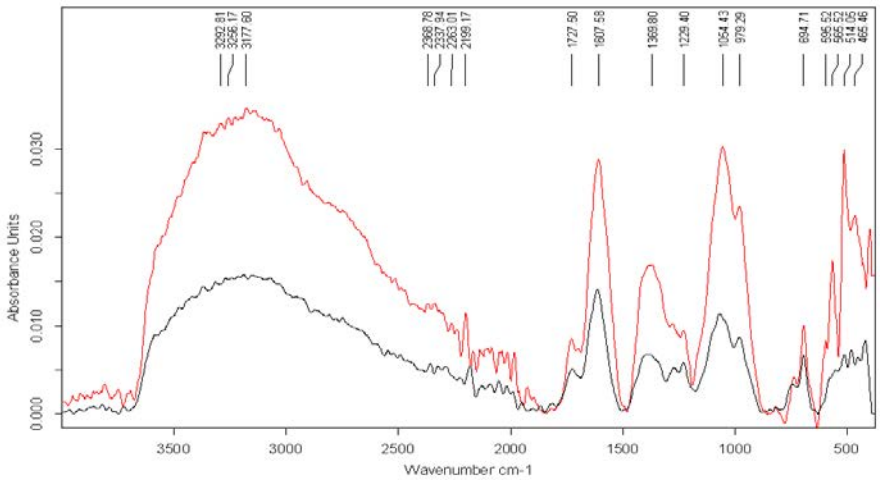
b) IR Spectrometry of GO and GO – C_{60}



a) GO



b) GO-C_{60}



c) GO and GO-C_{60}

Figure 4 : IR spectrum of products of oxidation of graphite and graphite with C_{60} : a) GO, b) GO-C_{60}, c) comparison of the spectrums a) and b

When preparing GO by oxidation methods FT-IR is usually indicated as a method for identification of the basic functional groups. In most cases the authors indicate vibration ranges for the given groups:

3000 – 3700 cm^{-1} ν (-COOH, -OH, H2O)

1850 – 1750 cm^{-1} ν (-C=O)

1650 – 1750 cm^{-1} ν (car boxy COOH)

1500 – 1600 cm^{-1} ν (sp2 C=C)

1280 – 1320 cm^{-1} ν (epoxides C-O-C)

At the same time, ranges that follow may contain the following vibrations:

1280 – 1500 cm^{-1}: ethers, epoxides, ketones, peroxides, benzoquinone

1100 – 1280 cm^{-1}: peroxides, ethers, ketones, lactones, anhydrides, epoxides, benzoquinones

900 – 1100 cm^{-1}: lactones, peroxides, hydroxyls, 1, 3 dioxane, anhydrides, epoxides, car boxy les-OH bond vibration at 3420 cm^{-1}, C=O bond vibrationat1720 – 1740 cm^{-1}.

The mutual comparison of spectrums we obtained for GO and GO-C_{60} is shown in Fig.4

The difference between the GO and GO-C60spectrums is in the ratio of the mutual adsorbances for the vibrations:

GO	1390	1274	1228 cm^{-1}
GO-C_{60}	1378	1278	1228 cm^{-1}

In thespectrumrange700 – 450 cm^{-1} the ads or bance of GO has a medium value while for GO-C_{60} it is high; in general, GO-C_{60} demonstrates higher adsorbances throughout the entire spectrum range.

c) Thermal Tests of GO and GO-C_{60} foils

Two prominent peaks were detected for samples of GO and GO-C_{60} foil son the DSC curve (Fig.5) which corresponded to exothermic processes. The first exothermic process was accompanied by a substantial drop of weight: for go by 43.6% and for GO–C_{60} it was even higher-51.1% (see Tab. 2).

In the case of GO the first exothermic process occurred at 190.9°Cwith the maximum at 225°C and thermal effect of 508.4 kJ/kg; in the case of GO-C_{60} the process started earlier, at 182.6°Cwith the maximum at 205°Cand the thermal effect was lower than for GO. The values of thermal effects in the individual temperature intervals are provided in Tab.3.The second exothermic effect in the case of GO has its maximum at 450°C, while for GO-C_{60} it was already at 390°C and the detailed shape of the curve was different (compare Figures5 and 6). For GO-C_{60} the weight loss during the second exothermic effect was lower than during the exothermic effect in the case of GO, which means a situation different from the first effect.

The weight losses until the first exothermic process are essentially the same for both the foils (ca. 20%) with mild end other mic effects, with a higher thermal effect for GO (anticipated dehydration). Also the overall thermal effect of decomposition is higher for GO-C_{60} foil by ca. 30% (see Tab.3).

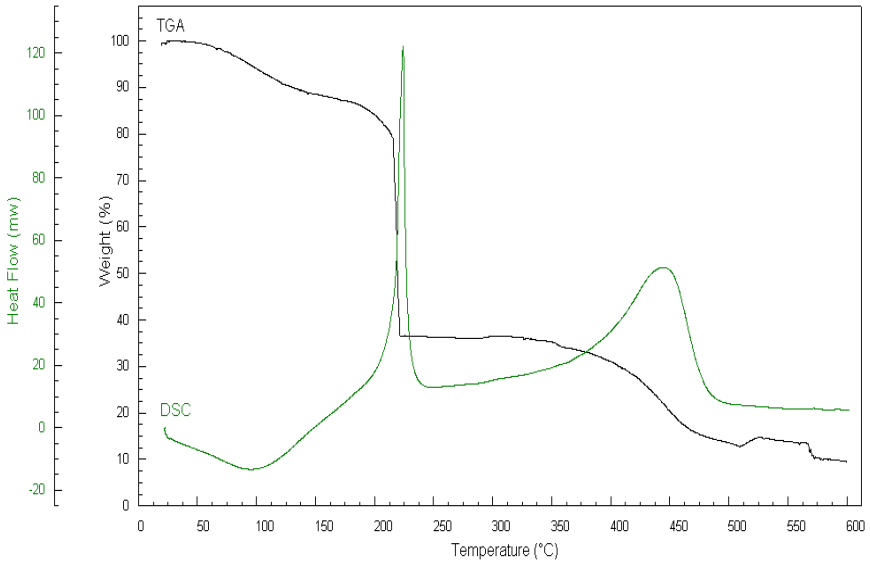


Figure 5 : Thermal analysis of a product of graphite oxidation– foil

(degradation medium: air, air flow : 20ml/min, temperature interval: 25-600°C, heating rate 10°/min, sample weight 10.0mg)

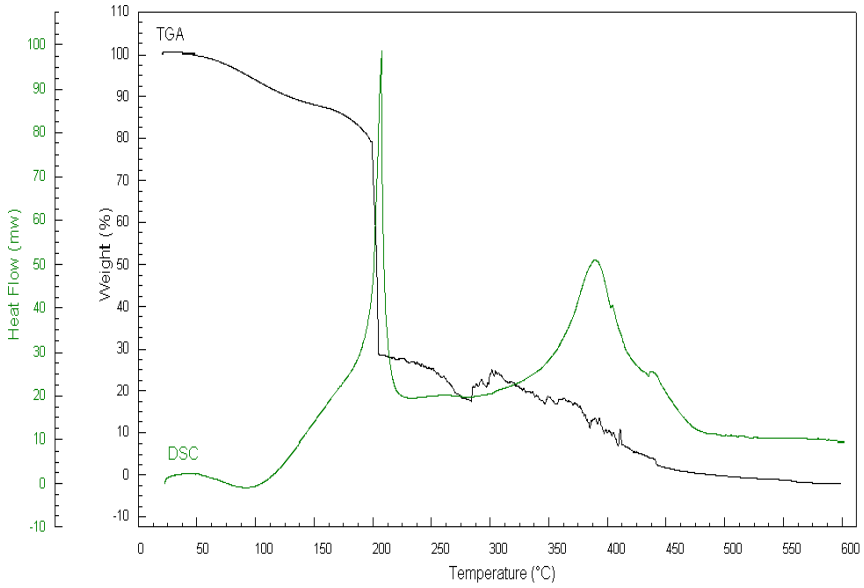


Figure 6 : Thermal analysis of a product of graphite and fullerene oxidation– foils

(degradation medium: air, air flow:20ml/min, temperature interval: 25-600°C,heating rate 10°/min, sample weight 10.0mg)

When performing the experiments we had some expectations about the course and results of there action. The functional groups expected on GO were the following: carboxyl, carbonyl, epoxide, hydroxyl and partly also lactone or sulfonic group. Fullerene C_{60} was expected to have the following groups on its molecule: - SO_3H, -OH, -NO_2, -ONO_2, epoxide group. Covalent bonds may form between those groups (esterification, dehydration, addition),as well as non-covalent ones – hydrogen bonds, π- π interaction, vander Waals forces. Also intercalation of a fullerene molecule may occur in the graphene-oxide space. Even breakage of a fullerene molecule in the conditions of oxidation cannot be excluded.

Table 2 : Division of the TGA curve into temperature intervals

Sample No.	Interval No.	Temperature range (°C)	Weight loss (%)
GO foils	1	25.0 – 142.4	11.0
	2	142.4 – 213.5	8.8
	3	213.5 – 222.3	43.6
	4	222.3 – 368.8	2.8
	5	368.8 – 473.0	18.1
	6	473.0 – 600.0	6.1
GO-C_{60} foils	1	25.0 – 87.1	4.4
	2	87.1 – 153.0	8.2
	3	153.0 – 197.0	8.0
	4	197.0 – 205.0	51.1
	5	205.0 – 281.3	10.8
	6	281.3 – 490.9	18.0

Table 3 : Parameters of the ongoing thermal processes (DSC)

Sample No.	Thermal process No.	Temperature range (°C)	ΔH (kJ/kg) *	H_{f1}(mW)	ΣΔH (kj/kg)
GO foils	1	25.0 – 154.1	874.6	15.8	-910.6
	2	190.9 – 241.1	-508.4	107.4	
	3	356.5 – 492.1	-1277.1	31.0	
GO-C_{60} foils	1	42.0 – 124.2	141.7	6.4	-1204.1
	2	182.6 – 221.5	-308.7	71.1	
	3	319.7 – 481.6	-1037.1	28.1	

**ΔH = thermal effect of the process based on the DSC curves*
(ΔH > 0…endothermic process, ΔH < 0…exothermic process)

One quoted work (Trzaskowki et al., 2013) deals with energy options of a combination of graphene with C_{60} on the condition that graphene surfaceis either defect-free or with defects. Chemical attachment of C_{60} on amonolayer of graphene is not possible for energy reasons. However, in presence of various defects on the grapheme C- layer, such as e.g. formation of 4- and 5-atomic rings, Stone-Wales defect and other types of defects (e.g. flower defect, octa-penta-, hepta-disrupted cyclic formations) may provide spaceorre active points for potential bonds with fullerene C_{60}.

One of the defects mentioned for the graphene structure is the so-called "adatom"–an adsorbed atom. It is a defect in which e.g. transition metal is adsorbedon the C lattice. Adsorption of transition metals changes physicochemical properties and biocompatibility of graphene or GO (Faye, 2012; Ne to, 2009). We have identified a similar defect in the prepared GO: it was e.g. an atom of Fe identified by EDAX in the GO structure (Fig.7).

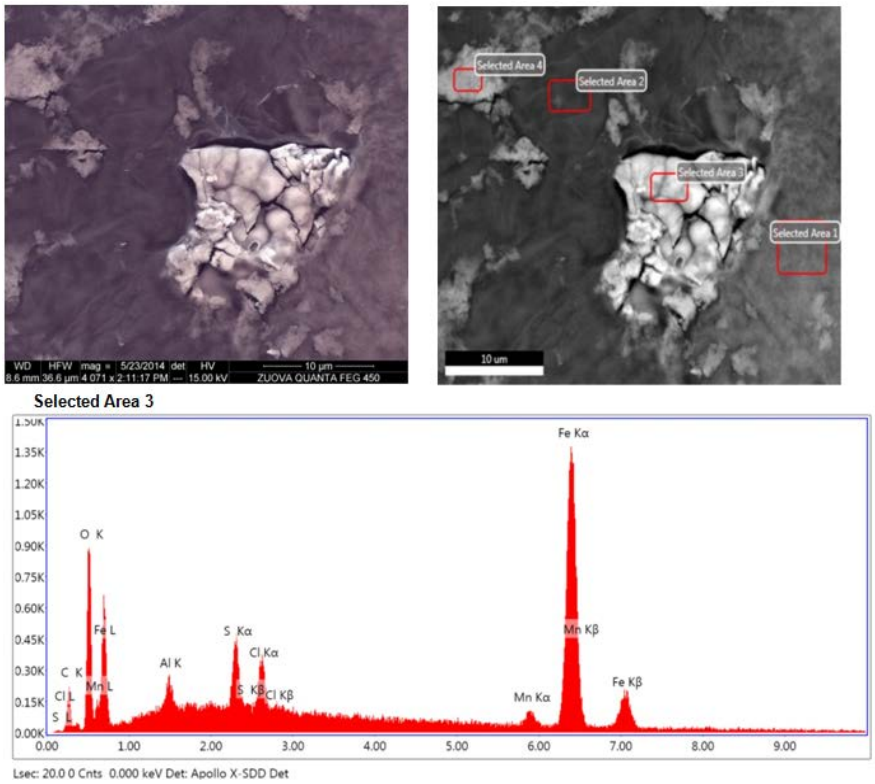


Figure 7 : Identification of a partly oxidize dadatom of Fe

We performed the so-called blank test to get some notion of how fullerene behaves in the oxidizing environment to which it is exposed jointly with graphite.

d) FT-IR Analysis of C_{60}-Oxi and Initial C_{60}

The measured IR spectrum of the initial fullerene C_{60} is provided in Fig.8. The main characteristic vibrations for C_{60} (Saeedfar et al., 2013) are 522 cm^{-1}, 573 cm^{-1}, 1159 cm^{-1}, and 1426 cm^{-1} and they are also a part of the C_{60}-oxi spectrum.

For C_{60}-oxi (Fig.9) we have identified an additional weak vibration in the absorption interval 1556-1644 cm^{-1} (probably C=C, C=O), 1000-1100 cm^{-1} (probably C-O-C epoxy, alkoxy, C-CO-C) and a medium vibrationat 702 cm^{-1} (probably a substituted romatic ring). Based on characteristic vibrations of functional groups we can probably exclude presence of the following functional groups on the C_{60}-oxi molecule:-OH, -COOH, -COOR, $-SO_3H$, $-NO_x$.

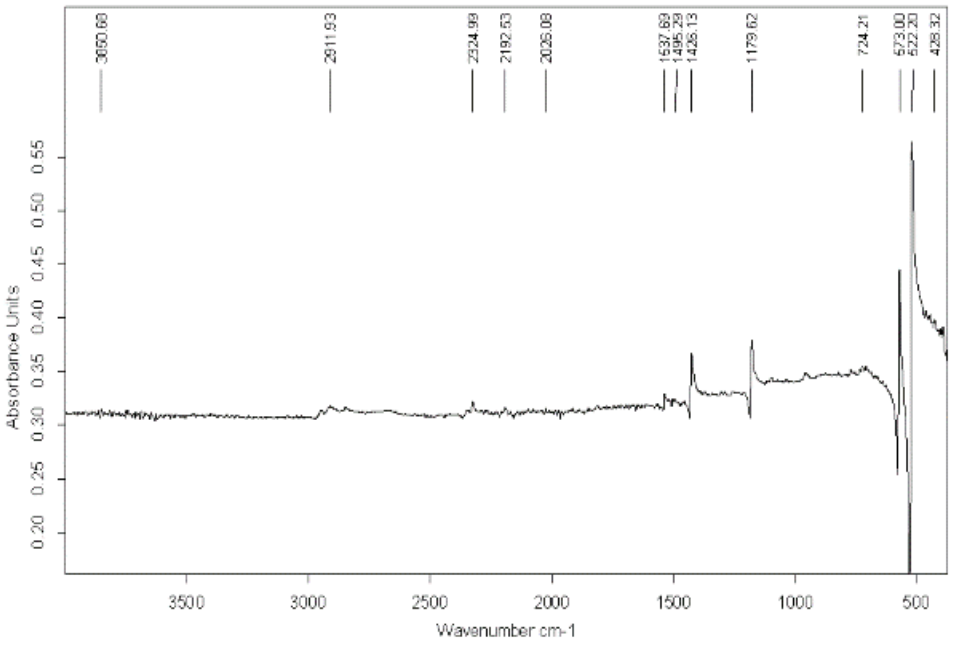


Figure 8 : IR-spectrum of C_{60}

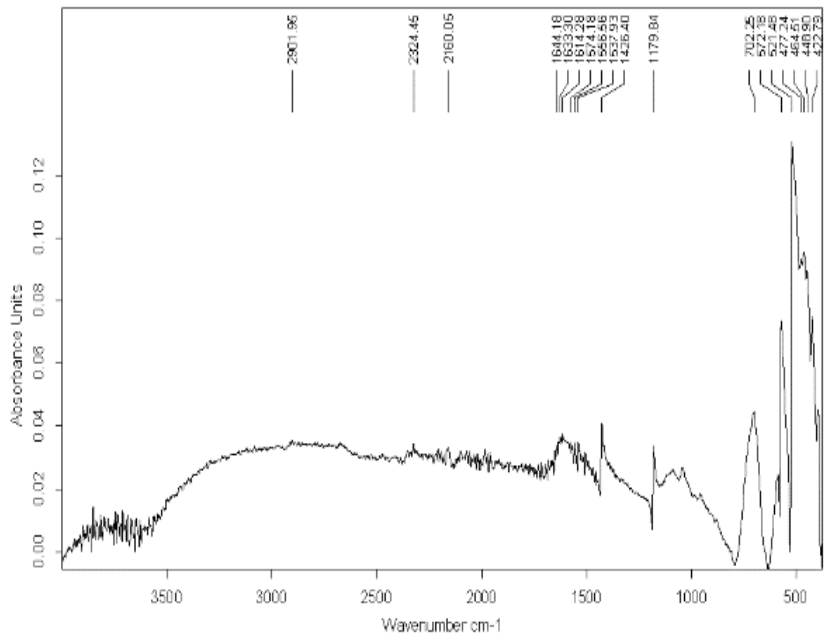


Figure 9 : IR-spectrum of C_{60}-oxi

e) Thermal Stability of C_{60}-Oxi and Initial C_{60}

In the measure demperature range, as indicated by the TGA and DSC curves (Fig.10-11) up to ca. 420°C,there were no thermal effects on either of the tested sample sand the weight loss of both the samples was comparable in units of percents (6.1% for C_{60} and 8.6%for C_{60}-oxi). In the temperature range of 420-600°C both the samples underwent exother micreactions, however the thermal effects were very different. The thermal effect of C_{60}-oxi was 20 times higher than that of the initial C_{60} (1947 kJ/kgfor C_{60}-oxi and 180 kJ/kg for C_{60}). The weight loss of C_{60}-oxi (56.7%) was twice bigger than that of C_{60} (28.8%).

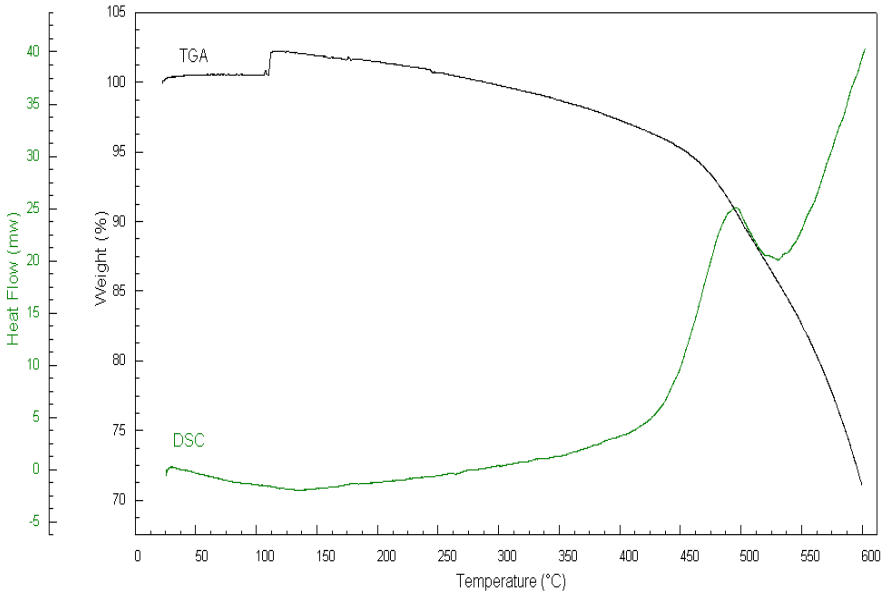


Figure 10 : Thermal analysis of C_{60}(degradation medium: air, air flow 20 ml/min, temperature interval 25-600°C, heating rate10°/min, sample weight11.21 mg)

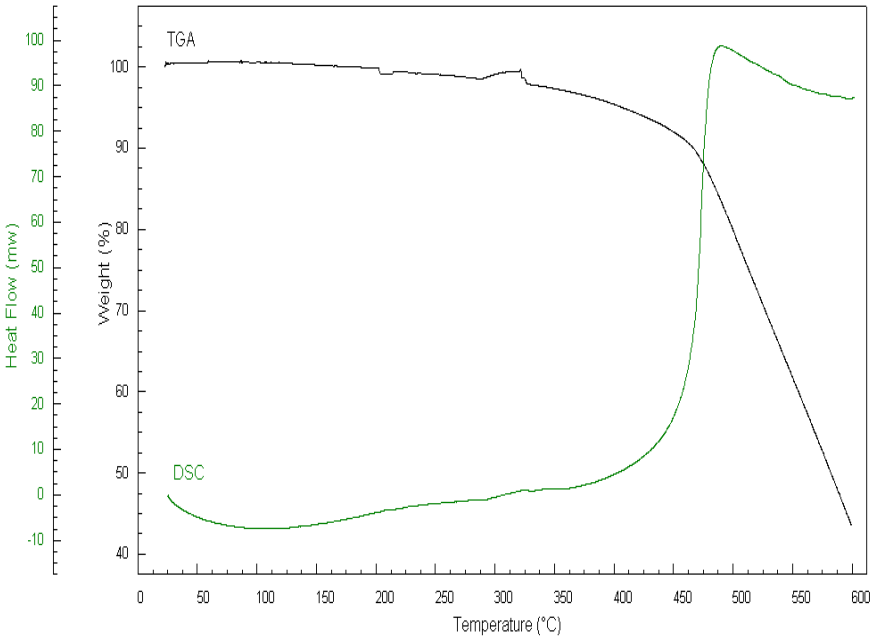


Figure 11 : Thermal analysis of C_{60}-oxi (degradation medium: air, air flow 20 ml/min, temperature interval 25-600°C, heating rate10°/min, sample weight 11.56 mg)

The resistance of C_{60}-oxi against thermal exposure was partly disrupted. We anticipate partial oxidation of the C-skeleton (IR-spectrum Fig.9) and thus disruption of its consistence (electron balance) which means that defects can appear in the carbon structure when the material is heated -e.g. the Stone-Wales defect (Kabiretal., 2011).

We have identified a similar reduction of resistance of the fullerene carbon skeletoninits bromo-derivative. After end other micdisruption of C-Brbond the C_{60} moleculefully (100%) decomposed at 420-550°C.

We are fully aware of the fact that this so-called" blind test" may not completely correspond to the oxidation process in presence of graphite or graphene-oxidein which a carbo-catalytic effect may apply (Navalon et al., 2014; Su and Loh, 2013).

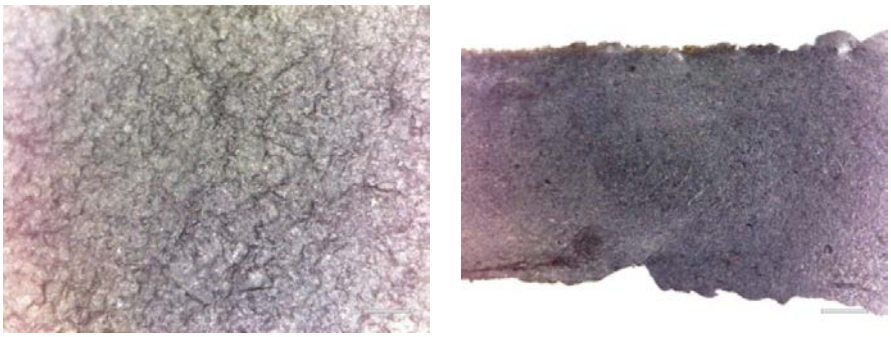
f) Reaction GO and GO-C_{60} with Cellulose

Nano cellulose – nano whiskers– can be prepared by hydrolysis of cellulose polymer (Bode son et al., 2006). Optimum conditions for the preparation depend on concentration of the employed acid

(H_2SO_4HCl), ratio of cellulose and acid, time of hydrolysis and on reaction temperature. Naturally, the result is also influenced by the type of the initial cellulose which can come from hard or soft wood, bamboo, sisal, cotton etc. All those factors in fluenceyield and size of the prepared cellulose nano fibers (loelovich, 2012; Li and Rage uskas, 2011). The prepared nano crystalline cellulose can bechemically modified, e.g. esterifed, carboxy lated oroxidized (Peng et al., 2011). It may be also used asa composite inpolymersor as a matrix for metal nano particles.

In our case we performed hydrolysis of methyl car boxy cellulose in presence of suspensions of GO and GO-C_{60}. We assumed that mutual interconnection may occur by a chemical reaction (e.g esterification, interconnection with C-O-C bond etc.) or physic chemical bond (e.g. hydrogenbonds). The foils prepared by vacuum filtration of the reaction product were subject to microscopic surface analysis, FT-IR, TGA and DSC analysis.

Morphology of the surfaces as shown by microscopic analysis (see Fig. 12) was different and it suggesteda potential method of interconnection between GO / GO-C_{60} and nanocellulose. The detailed morphology (electron microscope) of the prepared composite foils is shown in Fig.13 and14.



a) b)

Figure 12 : Roughmorphology of the foil surfaces after the mutualre action of GO-C_{60} (a) and GO (b) with cellulose

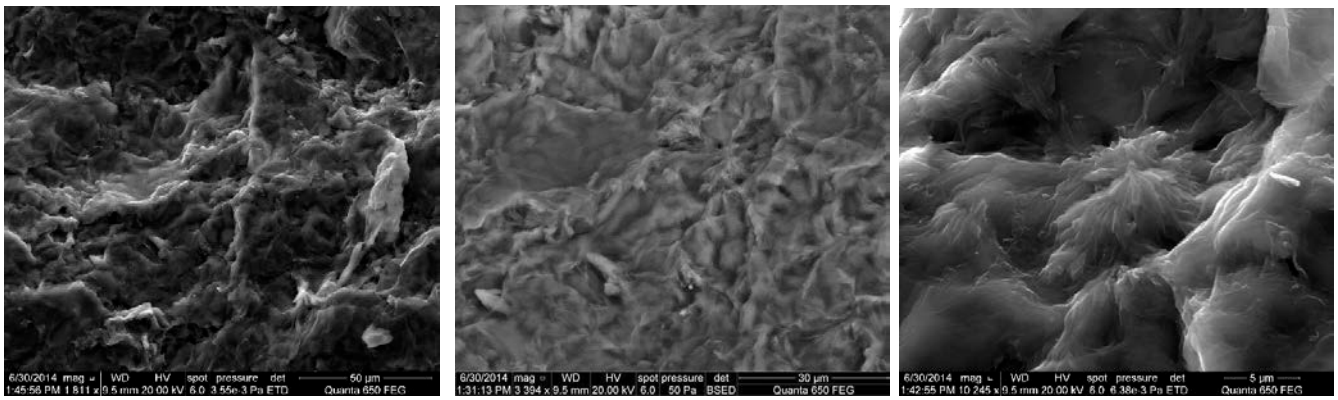


Figure 13 : SEM of GO-C_{60} foils with cellulose: scale 5 μm, 30 μm and 50 μm

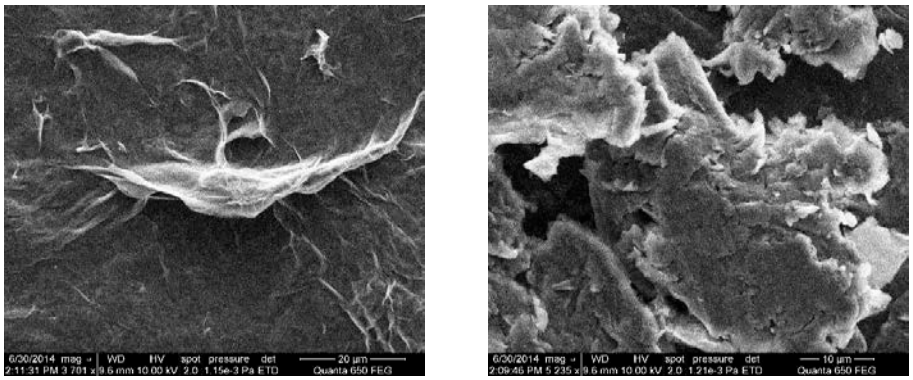
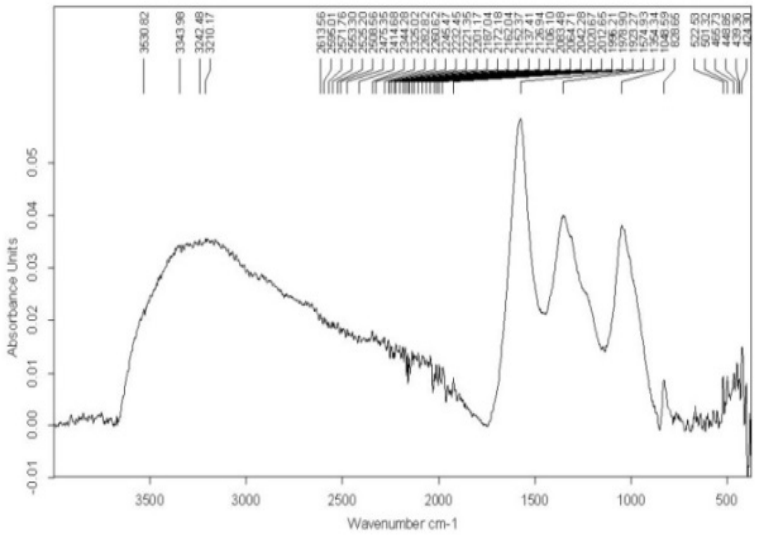


Figure 14 : SEM of GO-cellulose foils: scale10 μm and 20 μm

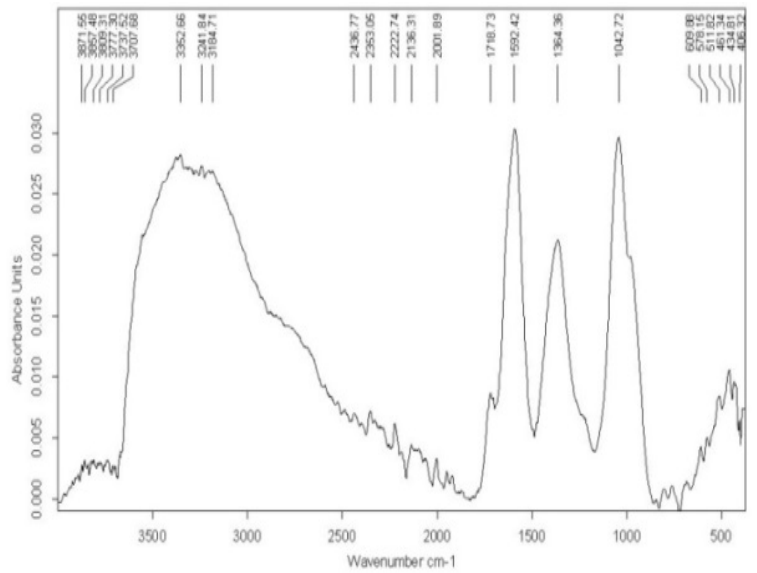
g) FT-IR Analysis of a Product of GO and GO_{60} Reaction with Cellulose (Nano cellulose)

We assume that in the first case the cellulose hydrolysis was only partial and whiskers of nano cellulose generated after the hydrolysis were of bigger size. At the same time, there was no reaction of C=O groups, unlike in the case of GO, where IR analysis of the product of reaction with cellulose identified no vibration at 1722 cm^{-1} ,which was present in the spectrum of the original GO. The other vibrations characterizing the groups C-O, C=C, C-O and C-O-C shifted their frequencies and also the mutual absorption ratios were different (Fig.15).

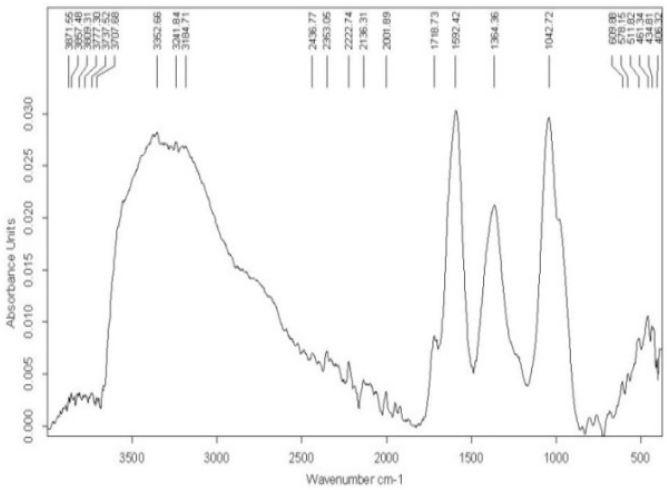
The same applies in respect to the initial GO, GO-C_{60} and cellulose (compare with the spectrums in Fig.4)



a) GO-C_{60}



b) GO



c) cellulose

Figure 15 : IR spectrums of products of reaction of GO-C_{60} (a) and GO (b) with cellulose and (c) the initial cellulose alone

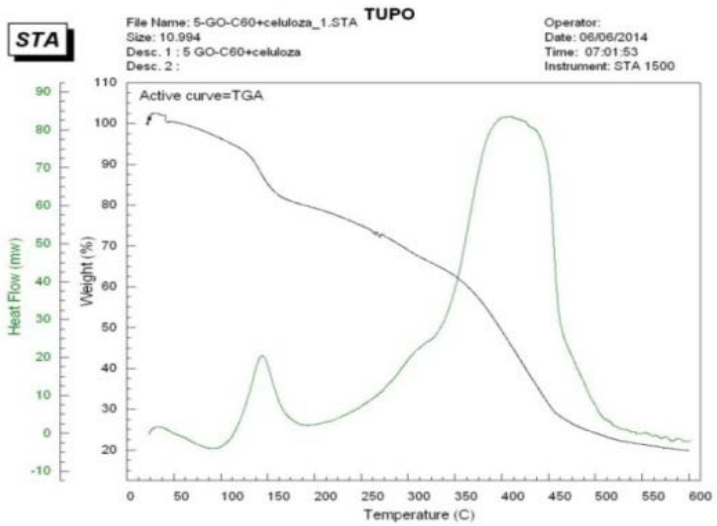
For the new products in both cases the IR spectrums did not contain the peaksat1278 cm-^{1} (1274 cm-^{1}) and 1228 cm-^{1} which in the original spectrums GO-C_{60} and GO had the assigned vibrations of epoxy groups.

Similar results, i.e. demonstration of deoxidizing (reduction) process, were described for the mutual reaction of GO with heparin (Wang et al., 2012), with a solution of cellulose in 1-butyl-3-methylimidazolium chloride (Peng et al., 2012) and with chi to san– starch (Rodrigues-Gonzales et al., 2012).

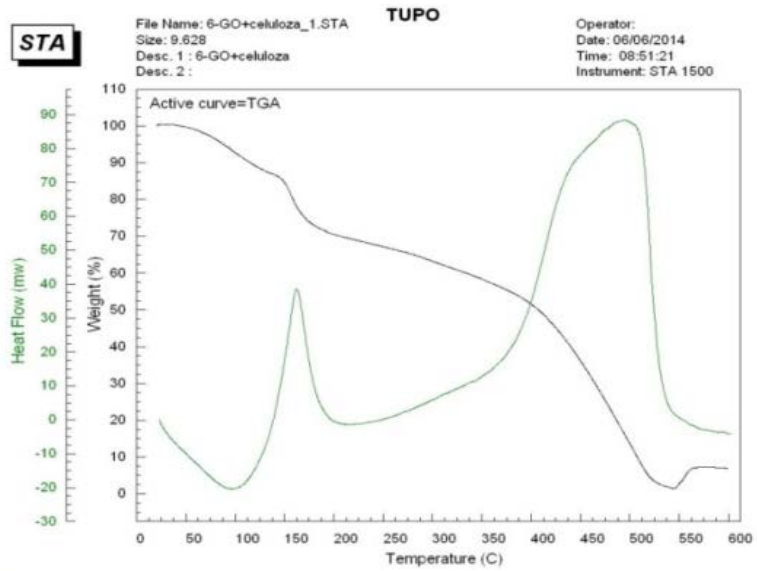
h) Thermal Stability of Products of GO and GO_{60} Reaction with Cellulose(Nano cellulose)

TGA curves of samples (see Fig. 16) of composites can be divided into several sections with different slopes, i.e. different weight loss rates. This division, including corresponding temperature intervals and corresponding weight losses, is shown in Tables 4 and 5, which provide parameters of the detected thermal processes on the DSC curve.

For composite samples the DSC curve showed one peak corresponding to an endothermic process and two peaks corresponding to exothermic processes. The second exothermic process was very substantial in both the samples. For the GO-C_{60}- cellulose sample the exothermic process started at 319.8°C and the peak area on the DSC curve was 3379.2 kJ/kg. Equally significant exothermic process in the GO-cellulose sample started at 341.5°C and the peak area on the DSC curve was 5261.4 kJ/kg.



a) GO-C_{60}- cellulose (degradation medium: air, air flow rate 20 ml/min, temperature 25-600° C, heating rate 10°/min, sample weight 9.0 mg)



b) GO - cellulose (degradation medium: air, air flow rate 20 ml/min, temperature 25-600° C, heating rate10°/min, sample weight 9.0 mg)

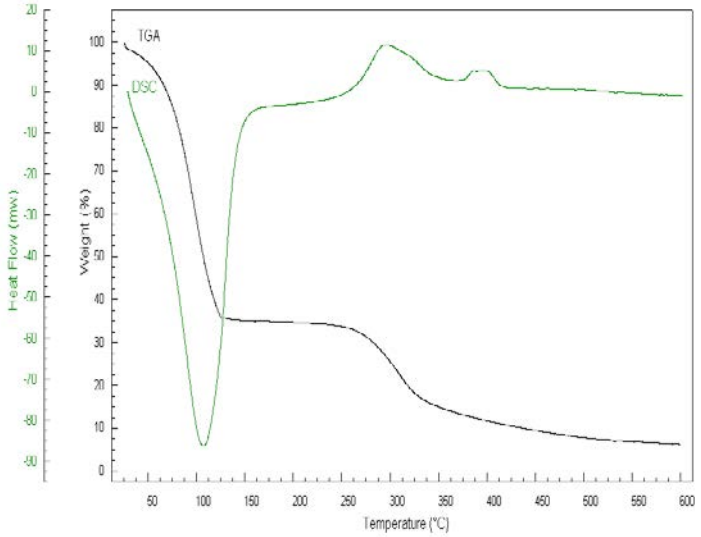


Figure 16 : IR spectrum of composites GO-C_{60}-cellulose a), GO-cellulose b), initial cellulose c) (degradation medium: air, air flow rate 20 ml/min, temperature 25-600° C, heating rate10°/min, sample weight 11.79 mg)

Table 4 : Division of TGA curves into temperature intervals

Sample No.	Interval No.	Temperature range (°C)	Weight loss (%)
GO-C_{60} – cel.	1	25.0 – 42.4	0.3
	2	42.4 – 123.9	6.9
	3	123.9 – 168.9	11.9
	4	168.9 – 347.8	18.5
	5	347.8 – 474.3	36.4
	6	474.3 – 600.0	6.8
GO – cel.	1	25.0 – 57.3	0.9
	2	57.3 – 120.8	10.1
	3	120.8 – 144.6	3.0
	4	144.6 – 180.6	13.2
	5	180.6 – 396.5	20.6
	6	396.5 – 522.6	48.3
	7	522.6 – 545.0	2.5
cellulose	1	25.0 – 62.9	8.5
	2	62.9 – 124.9	55.6
	3	124.9 – 265.8	3.3
	4	265.8 – 333.4	15.8
	5	333.4 – 600.0	10.5

Table 5 : Parameters of the ongoing thermal processes (DSC)

Sample No.	Thermal process No.	Temperature range (°C)	ΔH (kJ/kg) *	H_{f1}(mW)	ΣΔH (kj/kg)
GO-C_{60} – cel.	1	31.6 – 115.3	159.9	5.7	-3470.2
	2	115.3 – 187.2	-251.1	18.7	
	3	319.8 – 531.0	-3379.0	59.2	
GO – cel.	1	25.0 – 133.1	757.5	17.7	-5085.1
	2	133.1 – 208.1	-581.2	41.7	
	3	341.54 – 557.2	-5261.4	76.8	
cellulose	1	25.0 – 154.0	3053.7	86.2	+2805.7
	2	247.9 – 344.8	-211.3	12.2	
	3	372.0 – 414.6	-36.7	2.3	

**ΔH = thermal effect of the process based on DSC curves (ΔH > 0…endothermic process, ΔH < 0…exothermic process)*

The comparison of thermal stabilities of the prepared GO and GO-C60composites with cellulose indicates that the foil prepared from GO is thermally more stable but its decomposition releases more thermal energy.

A principle difference can be found when we compare thermal stability (weight loss) of the initial foils of GO and GO-C_{60} and thermal stability of their composites with cellulose. The weight loss in the temperature interval 25-220°C was 63% for GO foil sand 72% for GO-C_{60} foils. For the composites the weight loss was 32% and 22% respectively, which means a major difference. A completely different is the thermal decomposition of the initial cellulose. The decomposition in the temperature interval 25-154°C is accompanied by an endothermic process with the thermal effect 3053.7 kJ/kg and the weight loss of the sample is 64%.

i) GO Reaction with ß- 1,3-1,6- D-Glucan

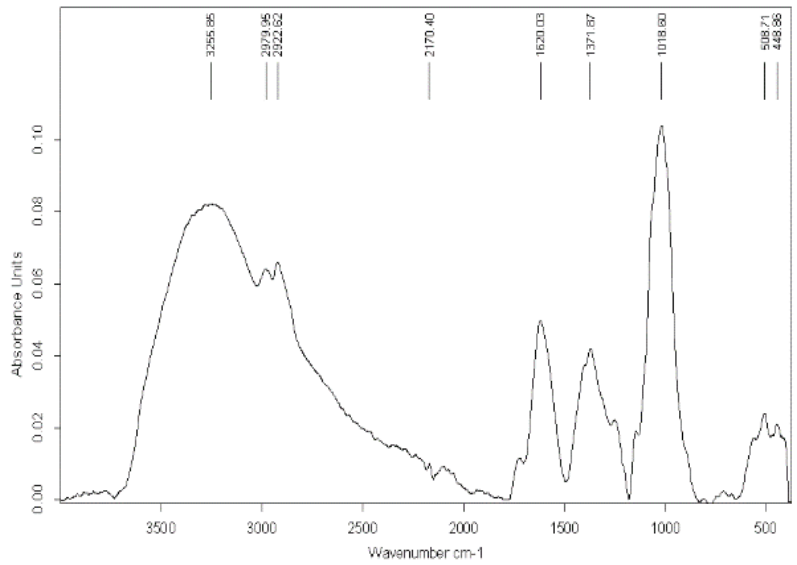
Glucans rank among homopolymsacharides, they have a long chain with only one structural component-glucose (hexose). Glucose in the chain is attached in the positions 1,3 and 1,6. Smaller chains branch off from the main chain. The structure of glucans is of extraordinary importance inactivation of the immunity system where branching of the side chains plays a majorrole.

ß-D-glucans are indigestible polysaccharides that occur in nature in sources such as cereal grains, yeasts, fungi, bacteria and algae. Biological effects of beta-glucans are manifested at several levels. The main role consists in activation of immunity system cells (macrophage) and they also performanti-carcinogenic, antimicrobial, antiviral and antialergic activities. Beta-glucans also have a radio protective effect–they deactivate free radicals (Chovancová and Šturdík, 2005).

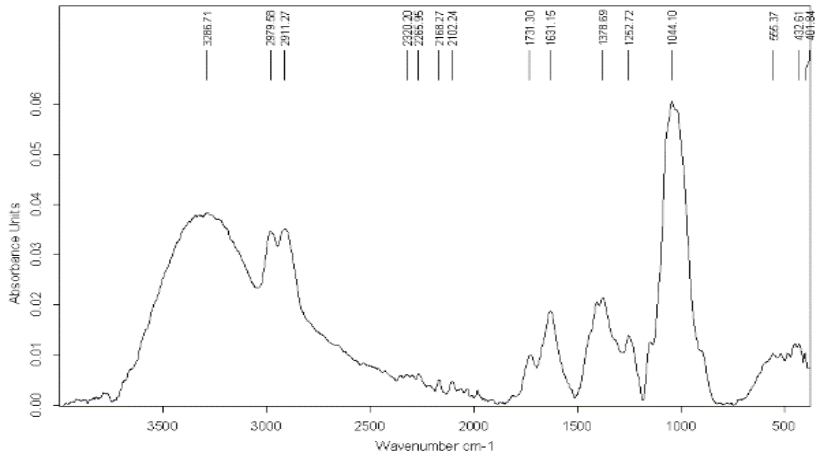
We performed reaction of beta-glucan with graphene-oxide under ultrasonification conditions with out sulfuric acid and in acid environment.

j) FT-IR Analysis of the Foils (GO-ßG and GO-ßGH+)

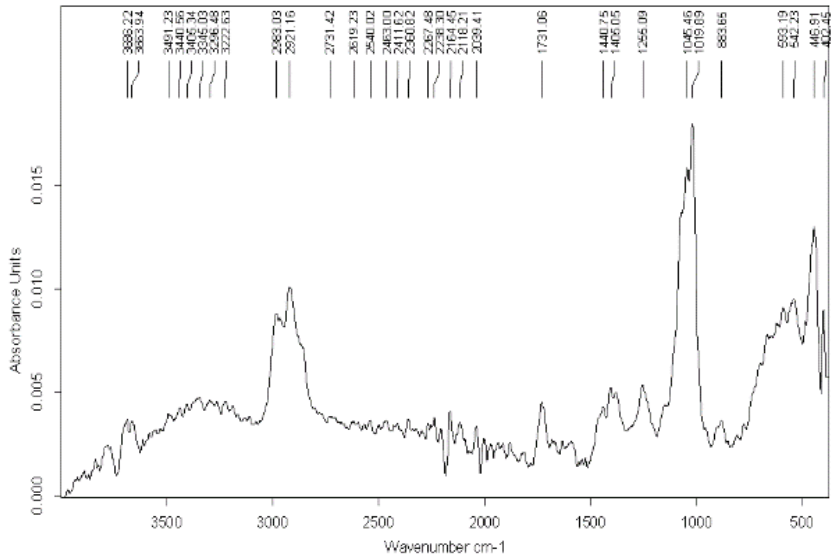
The obtained spectrums of the products are shown in Fig.17from left to right: GO-ßG, GO-ßGH+ and initial ßG. In the last picture d) the three spectrums are compared.



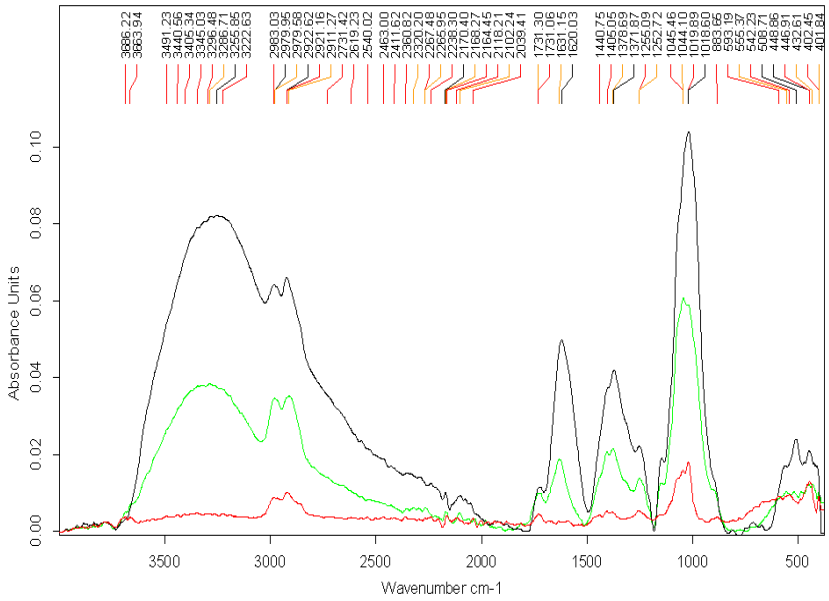
a) GO-ßG



b) GO-ßGH+



c) ßG



d) Combined spectrums. a) black + b) green + c) red

Figure 17 : IR spectrums of the reaction products: a)GO-ßG , b) GO- ßGH+ ,c) ßG, d) comparison of the spectrums shown ina + b + c

At the first sight the IR spectrums shown above are similarbuta more detailed inspection of the GO-ßG and GO- ßGH + spectrums shows differences in absorbance values, mutual ratio of peak intensity and in shifts of frequencies of decisive bond vibrations, e.g.:

	GO-ßG	GO- ßGH$^+$
C-O-C	1018 cm^{-1}	1044 cm^{-1}
-OH	3255 cm^{-1}	3286 cm^{-1}
-C=C-	1620 cm^{-1}	1631 cm^{-1}

The values of the initial GO are: C-O-C 1068 cm^{1}, 9.79 cm-^{1}, -OH 3149-3186 cm-^{1}, -C=C- 1613 cm-^{1}.

k) Thermal Stability of the Product (GO-ßG, GO-ßGH+) and Initial ßG

The measured results in a graphic form are provided in Fig.18-20 and interpretation of the TGA and DSC curves is provided in Tables 6-7.The DSC curve of ßG features one peak corresponding to an end other micthermal process with minimum weight loss and prominent, partly overlapping peaks that correspond to exothermic thermal processes which start at 257°C and are accompanied by a significant loss of the sample weight.

A common characteristic of both the prepared composite products is that their weight loss curves (TGA)can be approximated with a line –continuous linear reduction of weight, unlike the step weight loss in case of ßG which was up to 70% (see Fig.18) and in case of GO up to 60% (see Fig.5). The products differ from each other by the number of exothermic effect sand the shift of the last exothermic effect by 42°C in favor of GO-ß G H+.

The overall thermal effect in the course of decomposition decreases from the initial ßG to the GO-ßGH+ composite (see Tab.2).

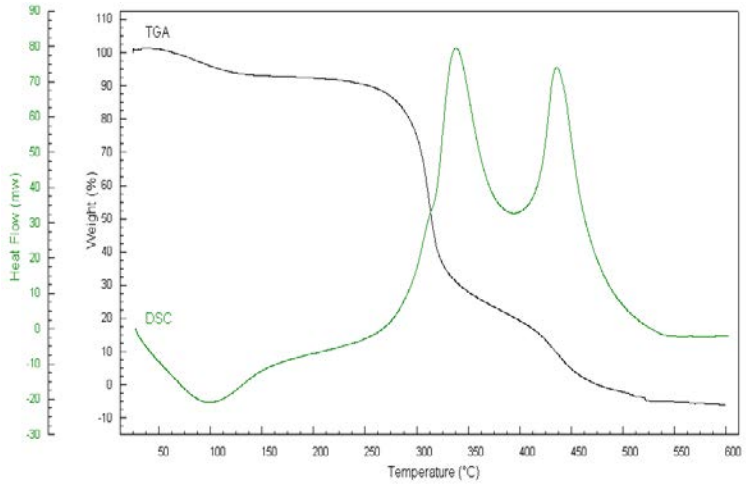


Figure 18 : Thermal analysis of ßG (degradation, medium: air, air flow rate 20 ml/min, temperature 25-600°C, heating rate10°/min, sample weight 11.03 mg)

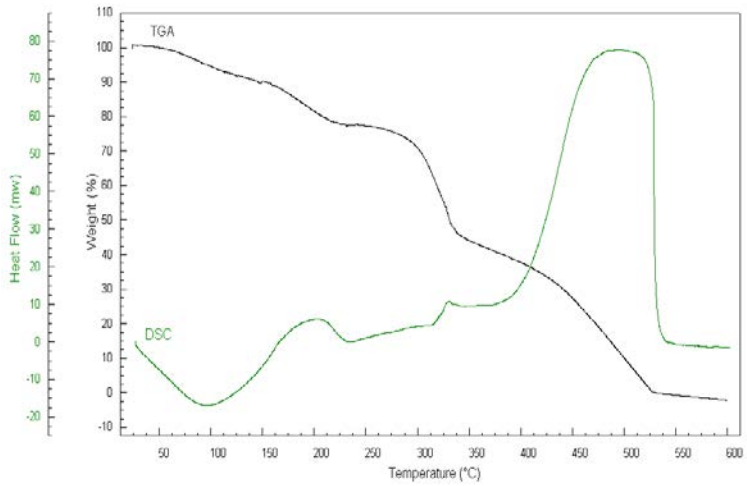


Figure 19 : Thermal analysis of GO-ßG (degradation medium: air, air flow rate 20 ml/min, temperature 25-600°C, heating rate10°/min, sample weight 11.47 mg)

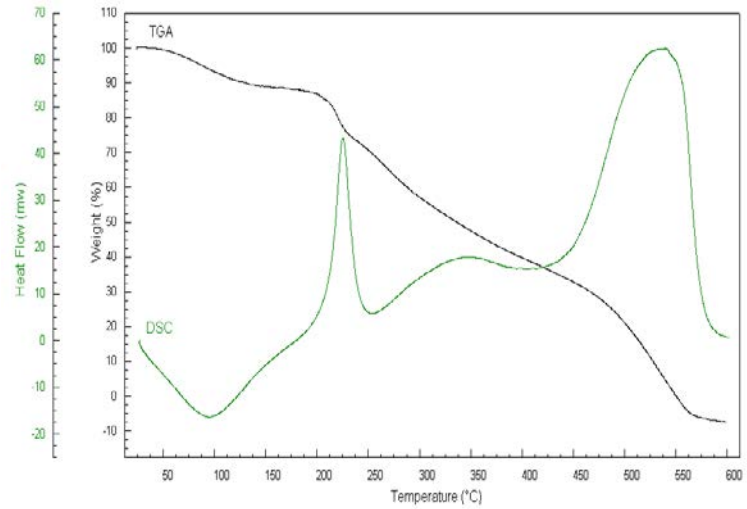


Figure 20 : Thermal analysis of GO-ßGH+(degradation medium: air, air flow rate 20 ml/min, temperature 25-600°C, heating rate10°/min, sample weight 10.77 mg)

Table 6 : Division of the TGA curves into temperature intervals [x]

Sample No.	IntervalNo.	Temperature range (°C)	Weight loss(%)
ßG	1	25.0 – 114.4	5.5
	2	114.4 – 278.1	8.6
	3	278.1 – 330.3	52.2
	4	330.3 – 412.7	17.0
	5	412.7 – 462.4	14.8
	6	462.4 – 475.9	14.8
GO- ßG	1	25.0 – 149.4	9.9
	2	149.4 – 225.5	12.3
	3	225.5 – 292.3	4.7
	4	292.3 – 337.9	26.7
	5	337.9 – 438.4	15.7
	6	438.4 – 529.8	30.7
GO- ßGH$^+$	1	25.0 – 124.2	9.9
	2	124.2 – 204.2	4.2
	3	204.2 – 230.8	10.9
	4	230.8 – 321.6	22.5
	5	321.6 – 480.9	25.9
	6	480.9 – 550.2	26.9

[x]*–the indicated intersections of tangents to the respective bends of the TGA curve*

Table 7 : Parameters of the thermal processes (DSC)

Sample No.	Thermal processNo.	Temperature range (°C)	ΔH (kJ/kg) *	H_{f1}(mW)	ΣΔH (kj/kg)
ßG	1	25.0 – 153.9	805.9	21.3	
	2	257.5 – 528.4	-4392.5	81.0	-3586.6
GO- ßG	1	25.0 – 157.0	825.3	16.9	
	2	157.0 – 228.8	-147.1	8.9	-2734.9
	3	311.0 – 356.5	-36.3	6.2	
	4	383.3 – 539.3	-3376.8	66.4	
GO- ßGH$^+$	1	25.0 -145.9	685.2	16.7	
	2	197.2 – 250.0	-313.5	38.2	-2084.7
	3	425.4 – 589.3	-2456.4	46.2	

ΔH = thermal effect of the process based on DSC curves (ΔH > 0…end other micprocess, ΔH □ 0⋯e xother micprocess)
Hfl =height of the peak of athermal processon the DSC curve in an absolute value related to the point corresponding to the beginning of the thermal process

III. Conclusion

Joint oxidation of graphite and fullerene C_{60} in the ratio2:1makes it possible to prepare a compound din form of compact foils but it has a lower thermal stability and its thermal decomposition is accompanied by thermal effects that are 30% bigger than effects produced by foils without fullerene. A method of attachment between oxidized graphite and fullerene has not been demonstrated. The prepared foil can also have other chemical and physicochemical properties thanks to the fullerene molecule (Trosh in et al., 2008).

Thermal decomposition of composites of GO and$GO\text{-}C_{60}$ with nano cellulose is accompanied by bigger thermal effects than the effects produced by the initial GO andGOC_{60}foils. The same applies also for ß-glucan in respect to GO. However, the thermal effect of the decomposition is lower than for ß-glucan alone.

The products we prepared were in the form of foils (membranes), with the exception of the "blank test" of fullerene oxidation. GO-foils (papers) are prepared by vacuum filtration of GO dispersion. This is the basic method of its preparation which we have also applied. We are fully aware of the fact that mechanical, electronic, chemical and biological properties and the related toxicological properties are affected by many factors which may have influenced our results of thermal stability measurements. The first factor is the method of GO preparation and thus there sultingratio of C/O and topology of the C-skeleton (its defects).The composition of the liquid phase and the concentration (Park et al., 2012) of the filtered suspension influence the foil thickness, as well as the filtration rate, and if the suspension is ultrasonificated then also its duration, temperature and power of the device play a role (Liao et al., 2011). Properties of foils are also influenced by some specific treatments, such as washing of foils with solution of MCl_2 (Ca, Ba, Mg), while the carbon layers are connected in a plane, across via dialdehyde (Hu et al., 2011), or its impregnatione.g. with Ti, Ag, Cu_2O (Chen et. al., 2011), expansion of the inter layer space (Zhu et al., 2012) etc.

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Oil Pipelines/Water Pipeline Crawling Robot for Leakage Detection/Cleaning of Pipes

By Shyam Lal Sharma, Abdul Qavi & Kamlesh Kumari
Al Falah School of Engineering and Technology, India

Abstract- Drive control system plays important roles in pipeline robot. In order to inspect the flaw and corrosion of seabed crude oil pipeline, an original mobile pipeline robot with crawler drive unit, power and monitor unit, central control unit, and ultrasonic wave inspection device is developed. Considering the limited space, a compact hardware system is designed based on an ARM processor with controllers. With made-to-order protocol for the crawl robot, an intelligent drive control system is developed. The implementation of the crawl robot demonstrates that the presented drive control scheme can meet the motion control requirements of the underwater pipeline crawl robot.

Keywords: robot, drive control system, RF technology, RFID device, antenna, decoder & encoder.

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Strictly as per the compliance and regulations of:

Oil Pipelines/Water Pipeline Crawling Robot for Leakage Detection/Cleaning of Pipes

Shyam Lal Sharma [α], Abdul Qavi [σ] & Kamlesh Kumari [ρ]

Abstract- **Drive control system plays important roles in pipeline robot. In order to inspect the flaw and corrosion of seabed crude oil pipeline, an original mobile pipeline robot with crawler drive unit, power and monitor unit, central control unit, and ultrasonic wave inspection device is developed. Considering the limited space, a compact hardware system is designed based on an ARM processor with controllers. With made-to-order protocol for the crawl robot, an intelligent drive control system is developed. The implementation of the crawl robot demonstrates that the presented drive control scheme can meet the motion control requirements of the underwater pipeline crawl robot.**

Keywords: robot, drive control system, RF technology, RFID device, antenna, decoder & encoder.

I. Introduction

Robots have become important over a wide range of applications--from manufacturing, to surgery, to the handling of hazardous materials. Consequently, it's important to understand how they work, and what problems exist in designing effective robots. A robot is an automatically guided machine which is able to do tasks on its own, almost always due to electronically-programmed instructions. Mobile robots have the capability to move around in their environment and are not fixed to one physical location. This project presents a dynamic model for a novel pipeline robot which obtain sits power from *12V* battery. The robot is designed to move both against and with the flowing fluid, which makes it different from conventional solutions, which can only move with the flowing fluid. This bidirectional capability makes it very valuable to many industries, especially the oil and gas industries. This robot includes several novel mechanical features, including novel designs for its chassis and, energy-dense power transmission to enable high-speed crawling.

Industrial ductwork has been widely used in metallurgy, petroleum, chemical engineering, water supply and other special professions. The formidable work environment makes pipelines easy to be eroded or fatigued which can lead to leaking accident, so the periodic maintenance and overhaul are necessary for industrial pipelines. Absence of fresh air makes it Impossible for humans to perform maintenance task.

Author α σ: Department of Mechanical Engineering, Al Falah School of Engineering and Technology, Fbd, Hr.
e-mail: shyambash2009@yahoo.in
Author ρ: Mechanical Design Engineer, Desein & Indure Pvt. Limited, New Delhi.

As maintenance of these pipelines is nearly impossible from outside we need a machine that can crawl inside these long pipes. The crawling robot can be wirelessly steered into the long pipelines. The embedded cameras will use the complete inside picture of a long pipeline, which would help us, detect and fix the leakages or any other technical problems. The above Robot can also be used for cleaning of the pipelines.

Currently, in-pipe robot with tether, which enables the robot to have the enough energy supplies and promptly make up the power loss, still has important application value owing to avoid carrying heavier energy devices, but the noticeable friction forces of tether restrict the traction force of robot, locomotion distance away from entrance, and the steering inside pipelines with elbows. Therefore, the development of autonomous in-pipe locomotion robot without tether becomes urgent, such that the robot can be adaptive to the work of long and complicate pipeline.

One of the key techniques to develop in-pipeline locomotion robot is electrical drive. More driving spots, more flexible action, lower power consumption and other special requirements are making the motor driving technique very challengeable. Based on simulation prototype of in-pipe robot driven by wheels for inspection the inner surface of seabed pipelines, this project focuses on the drive control system of its engineering prototype without tether, including design drive control system based on engineering requirements, hardware design of the control system, intelligent crawling control and experiments.

II. Objective

Although the appearance and the capabilities of robots vary vastly, all robots share the feature of a mechanical, movable structure under some form of control. Designing a robot chassis will be the first objective of this project. The Control of robot involves three distinct phases: - perception, processing and action. Generally sensors/or command receivers are preceptors mounted on the robot, processing is done by the on-board microcontroller, and the task is performed using motors or with some other actuators. We need to design a wireless circuit which helps robot climb on the pole, can be controlled using wireless technology like RF technology. We need to design a controller and a power transmission system. We also need to design a wireless video transmission system.

III. Motivation

The making of a pipeline crawling robot and planning its responses against the signals captured from its onboard video camera supposes an excellent incentive for student's learning. The observe defect is that once the problem is stated, the student himself sets out challenges to surpass his talent and abilities in order to expand the robot with new sensors and actuators that may improve its behavior.

Compared to other conventional mobile robots used for teaching, Oil pipeline crawling robot incorporates sophisticated and diverse elements, to design the circuit, chassis and remote control via cable or via RF. It is a complete package for learning in the design of hardware-software systems, including the design of communication interfaces, adapters, power circuits, signal transmitters etc.

IV. Project Overview

For years, engineers and owners of pressurized pipelines have been seeking better ways to monitor the condition of buried pipes. In lieu of exposing pipes for inspection, pipeline owners have used various types of remotely controlled probes, cameras, and other devices to obtain information about underground pipes. The oil and gas industries have largely led the charge, driven by regulations and risks associated with catastrophic failures and explosions.

Recent developments indicate that petroleum industry techniques can be tailored for municipal applications, such as water distribution pipelines. Robotic systems are offering new capabilities in obtaining real-time data, capturing the attention of budget-conscious owners of aging infrastructure systems. The July explosion of a steam pipeline in New York City provided a wake-up call that buried utility pipes cannot be considered out of sight, out of mind.

Here we designed a robot that can be used for analyzing the conditions of the pipelines and clean them if required. The main components used in this are: DC motor, motor driver, RF transmitter& receiver, 12V battery .Basically, the robot is controlled by a RF based remote that send RF signals to the receiver i.e. installed on the robot.

Our prototype of 'Pole Climbing Robot' has the capability to move inside the pipes and perform the desired task smoothly. For moving inside pipes, robot wheel based chassis is developed. Enough pressure should be applied on wheels to create sufficient friction to hold the robot on its place.

V. Description of Working

The major concepts/components used in this project are RF technology, motor driver, Chassis-designing, power circuit and power transmission etc. We designed an RF based control system to control Robot. The signal get generated and transmitted with the help of antenna, provided on it. Now the receiving section which we already installed on the robot will receive the signals and send the received signals to the motor drive, on the basis of which motor driver will drive the robot and robot climb on the pole.

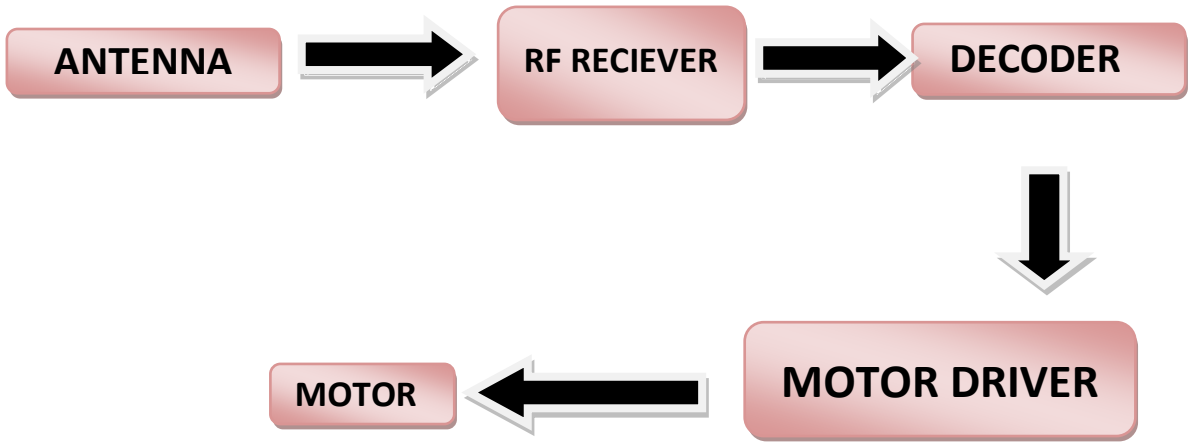
Radio Frequency is a flexible technology that is convenient, easy to use, and well suited for automatic operation. It combines advantages not available with other technologies. It can be supplied as read-only or read/write, does not require contact or line-of-sight to operate, can function under a variety of environmental conditions, and provides a high level of data integrity. In addition, because the technology is difficult to counterfeit, RFID provides a high level of security. Radio waves transfer data between an item to which an RFID device is attached and an RFID reader.

The device can contain commands for robot. RF technology uses frequencies within the range of 50 kHz to 2.5 GHz. An RFID system typically includes the following components:

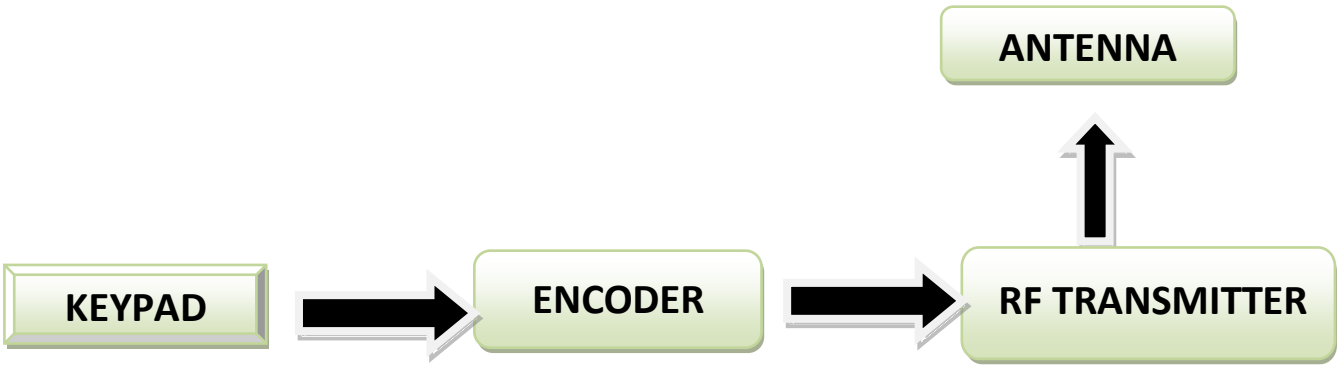
- An RF devices (transponder or tag) that contains data about an item/location
- An antenna used to transmit the RF signals between the reader and the RFID device
- An RF transceiver that generates the RF signals
- A reader that receives RF transmissions and passes the data to a host system for processing. To control robot a remote is designed which has a number of switches on its control board. As soon as you press any button it transmits signals that are received by receiving section then it decodes it and on the basis of signals, motion takes place in the robot like forward, backward. Robot receives this signal with the help of antenna present on the head of the robot.

VI. Block Diagram

➢ *Device:*



➢ *Remote Control:*



VII. Description

The constructed robot is designed in such a way that it can move in pipe very efficiently having a given range of diameter. The robot is RF controlled. The complete working of RFI based system can be divided in the following blocks for easier understanding:

a) Components list

- HT12 E
- HT12D
- L293D
- DC MOTOR
- RF MODULES(434 MHz)

i. *RF Transmitter Unit*

This 4 bit data is essentially a RF transmitter that transmits encoded signals. It can be further classified into following parts:

❖ *DATA 4 bit through switch*

This part is unique for every RF. It creates a 4-bit data that is used to identify when the data is read by RF reader.

❖ *ENCODER*

This part converts the data into an encrypted data that can be transmitted over RF channels. The encoder used here is HT12E which can be used to encrypt 4 bit data. The encrypted data is a serial digital signal.

❖ *TRANSMITTER*

This part takes the encrypted data from transmitter and transmits it in form of Radio Frequency. The transmitter used here is ASK 434 MHz RF-TX modules.

❖ *POWER SUPPLY BLOCK*

This consist a 12V power supply source and a power regulator (7805) to get 5v power supply. This 5v supply drives the transmitter and the encoder.

ii. *RF data RECIEVER*

This reader is basically a RF receiver that receives encoded signals decodes them and. It can be further classified into following parts:

❖ *RECIEVER*

It receives the encrypted data in form of RF waves and converts it into electronic signals. The receiver used here is ASK 434 MHz RF-Rx modules.

❖ *DECODER*

This part decrypts the data to yield the 4-bit data. This data is fed into the circuit. HT12D has been used here which is compatible with HT12E module. The output is the 4 bit data format.

❖ POWER SUPPLY BLOCK

This consist of a 12V power supply source and a power regulator (7805) to get 5v power supply.

b) Component Details

i. *RF Module*

Radio Frequency Module is an integral part of boarder security system together with a control module or unit and an antenna it is used for wireless identification. Main tasks of the RF module are to send an energizing signal via the antenna. The RF module delivers a digital data stream and a clock signal for further processing to its control unit or module.
Furthermore a field strength dependent digital output is available for synchronization purposes. The RFM is tuned to resonance with the antenna by adjusting the inductance of the tuning coil at the RFM's output stage.
RF Module can be categorized into two parts:

- Transmitter
- Receiver

a. TRANSMITTER

This wireless data is the easiest to use, lowest cost RF link we have ever seen! Use these components to transmit position data, temperature data, and even current program register values wirelessly to the receiver. These modules have up to *500 ft range* in open space. The transmitter operates from 2-12V. The higher the Voltage, the greater the range - see range test data in the documents section. We have used these modules extensively and have been very impressed with their ease of use and direct interface to an MCU. The theory of operation is very simple. This is an ASK transmitter module with an output of up to 8mW depending on power supply voltage. The transmitter is based on SAW resonator and accepts digital inputs, can operate from 2 to 12 Volts-DC, and makes building RF enabled products very easy.

Features

- 434 MHz Transmitter Operation
- 500 Ft. Range - Dependent on Transmitter Power Supply
- 2400 or 4800bps transfer rate
- Low cost
- Extremely small and light weight

b. RECEIVER

This receiver type is good for data rates up to 4800bps and will only work with the 434MHz transmitter. Multiple 434MHz receivers can listen to one 434MHz transmitter. This wireless data is the easiest to use, lowest cost RF link we have ever seen! Use these components to transmit position data, temperature data, even current program register values wirelessly to the receiver. These modules have up to 500 ft range in open space. The receiver is operated at 5V. We have used these modules extensively and have been very impressed with their ease of use and direct interface to an MCU. The theory of operation is very simple.

Features

- 434 MHz Operation
- 4800 bps transfer rate
- Low cost
- Extremely small and light weight

ii. *MICRO SWITCH*

A micro switch, also known as snap-action switch, is a generic term used to refer to an *electric switch* that is actuated by very little physical force, through the use of a tipping-point mechanism. They are very common due to their low cost and durability, greater than 1 million cycles and up to 10 million cycles for heavy duty models. This durability is a natural consequence of the design. Internally a stiff metal strip must be bent to activate the switch. This produces a very distinctive clicking sound and a very crisp feel. When pressure is removed the metal strip springs back to its original state. Common applications of micro switches include the door *interlock* on a *microwave oven,* leveling and safety switches in *elevators, vending machines,* and to detect paper jams or other faults in photocopiers. Micro switches are commonly used in tamper switches on gate valves on fire sprinkler systems and other water pipe systems, where it is necessary to know if a valve has been opened or shut.

The defining feature of micro switches is that a relatively small movement at the actuator button produces a relative large movement at the electrical contacts, which occurs at high speed (regardless of the speed of actuation). Most successful designs also exhibit *hysteresis,* meaning that a small reversal of the actuator is insufficient to reverse the contacts; there must be a significant movement in the opposite direction. Both of these characteristics help to achieve a clean and reliable interruption to the switched circuit.

The first micro switch was invented by *Peter McGall* in 1932 in *Freeport, Illinois*. McGall was an employee of the Burgess Battery Company at the time. In 1937 he started the company MICRO SWITCH, which still exists as of 2009.

The company and the *Micro Switch* trademark have been owned by *Honeywell Sensing and Control* since 1950.The trademark has become a widely used description for snap-action switches. Companies other than Honeywell now manufacture miniature snap-action switches.

Micro switches are applied in appliances, machinery, industrial controls, vehicles, and many other places for control of electrical circuits. Micro switches are usually rated to carry current in control circuits only, although some switches can be directly used to control small motors, solenoids, lamps, or other devices. Micro switches may be directly operated by a mechanism, or may be packaged as part of a pressure, flow, or temperature switch, operated by a sensing mechanism such as a *Bourdon tube*. A motor driven cam and one or more micro switches form a timer mechanism. The snap-switch mechanism can be enclosed in a metal housing including actuating levers, plungers or rollers, forming a *limit switch* useful for control of machine tools or electrically-driven machinery.

iii. *MOTOR DRIVER*

Here we used L293D to drive the motors. whatever signals it receives from the on the basis of that it will drive the motors.

a. DC MOTOR

A direct current (DC) motor is a fairly simple electric motor that uses electricity and a magnetic field to produce torque, which turns the motor. At its most simple, a DC motor requires two magnets of opposite polarity and an electric coil, which acts as an electromagnet. The repellent and attractive electromagnetic forces.

VIII. Background of the Robot and Hardware Design of The Control System

a) Introduction of the crawling robot

In order to inspect the seabed petroleum pipelines of Oil Field, a robot is developed. The overall length of pipeline is long range, and the inner diameter of the pipe is greater than 8 inches. In order to locate the pipe defects, the inspection process is divided into two steps: firstly, the oil differential pressure drive type and the supersonic inspection principles are utilized to realize the on-line inspection; secondly, based on the information of first step, using the in-pipe robot completes the real-time localization inspection. This project is belonged to the second step, and the main technique indexes are:

- Normal crawling speed is optimum.
- Driving wheels can adapt to various diameters of the pipelines automatically.
- The robot can operate in water with 2MPa pressure safely.

Based on the design indexes, motors and their drivers should be smaller and better performance. As shown in Figure 1, the in-pipe robot inspection system contains ten units, including crawler unit, drive unit, central controller unit, battery unit and ultrasonic inspect unit, etc. The drive control system receives motion commands and drives the robot forward and backward. The magnets provide the torque that causes the DC motor to turn.

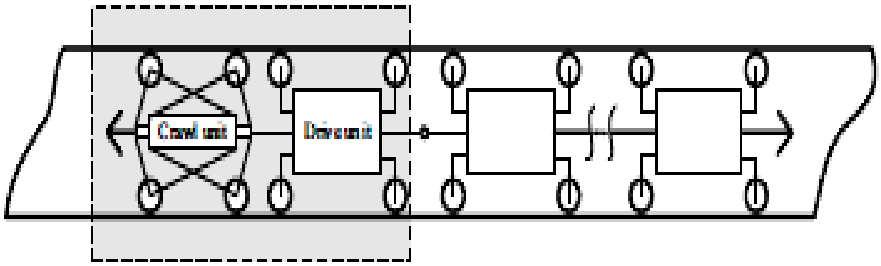


Figure 1 : Overall structure of the in-pipe craw1 robot

b) Hardware design of the control system

The control system has an advantages of high communicating efficiency, reliable, stable, and easy to set up, which makes it feasible to the 4D (Dull, Dirty, Difficult and Dangerous) workplace like in-pipe inspection. Considering space limitation and low energy cost, an ARM processor. The drive control system is shown in Figure 2. In order to realize the locomotion control of the robot, the peripheral AD, DA, DI and DO are designed to control the motors. Real-time current detection for the motors guarantees the safe operation of the motors and intelligent motion control of the robot.

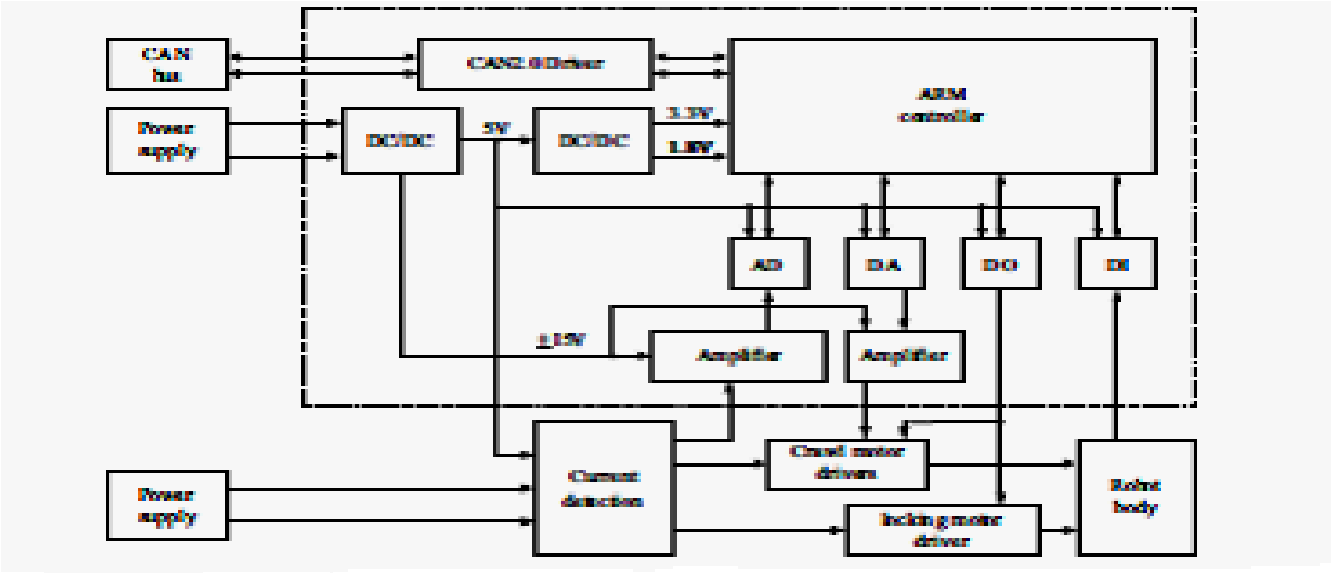


Figure 2 : Hardware connection of control system

c) Crawling control

The fundamental function of crawling control system is communication between crawling units and central controller. Generally, crawling drive unit executes the command from central controller and reports its operational results in time. The software of drive control system is developed with ADS1.2 and mainly consists of CAN communication, locomotion control, and current detection. Figure 3 shows the workflow of drive control of the robots.

- CAN communication: When the system is electrified, drive control system begin self-testing, and successful self-testing result will be reported to the central controller which means the following work can run, otherwise system default. Among the following work, drive control system will keep communicating with other nodes to guarantee its teamwork.
- Locomotion control: Locomotion control is the key job of drive control system. Actions of the robots include locking, unlocking, micro-locking, micro-unlocking, crawling forward, crawling backward, pause crawling, stop crawling, accelerating and decelerating. The locking state of the mechanism is related to the maximum drive force, so the micro-locking is designed to insure the best locking of the mechanism. After the successful locking of the mechanism, drive control system will carry out the appropriate locomotion. Among the crawling job, soft start-up, slope speed setting, and soft stoppage are considered to make the motion more reasonable.
- Current inspection: Current detection for locking motor is helpful to realize the best locking gesture, protect motors, and achieve the three crawl modes, namely, unification drive mode, grouping drive mode and independent drive mode. The three drive models are design to make the robot adapt to the straight, curved and slope pipelines.

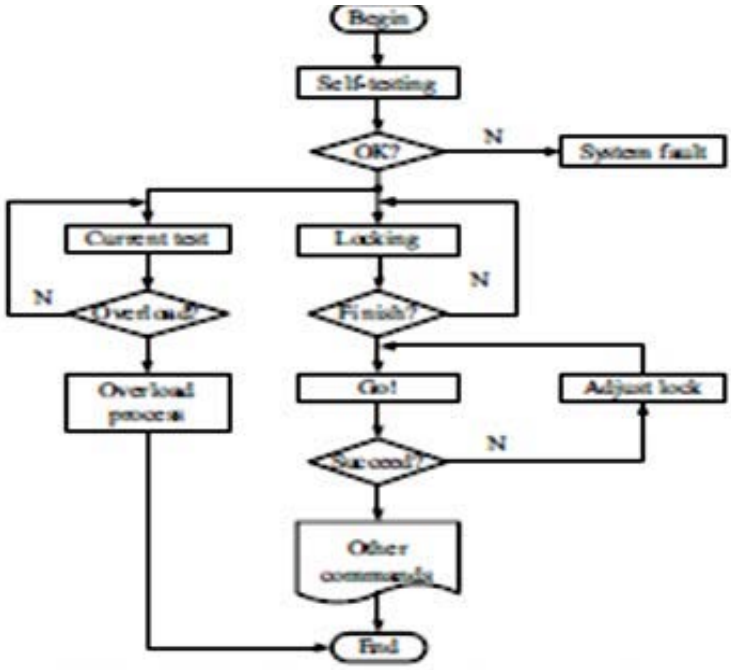


Figure 3 : Workflow of the crawling control

IX. Applications

This locomotion can be used in various applications; some of them are as follows:

- Pipeline maintenance work
- Fixing of any leakages
- Cleaning of the pipelines
- Maneuvering in hazardous environment

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Fullerenec$_{60}$, Graphene-Oxide and Graphene-Oxide Foil with Fullerene and their Bromination

By Klouda Karel, Zemanová Eva, Friedrichová Romana & Weisheitová Markéta

VŠB-Technical University of Ostrava, Czech Republic

Abstract- A direct reaction with liquid bromine was used to prepare bromo fullerene C_{60} Br_{14-18}. The brominated derivative reacted with previous lyprepared graphene-oxide (hereinafter GO), according to a method described by Hummer. The same method was used to oxidize graphite alone. The prepared graphite fullerene foil was brominated with liquid bromine and the graphene-oxide foil was reacted with bromo fullerene. FTIR analysis of all the obtained products was performed and also TG Aanalysis to investigate particularly their thermal stability. The brominated products demonstrate lower thermal effects when thermally decomposed which is caused by the retarding ability of bromine.

Keywords: liquid bromine, fullerene c_{60}, graphene-oxide foil, graphene-oxide foil with fullerene, brominated fullerene.

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I. Introduction

Graphite is an allotropic modification of carbon with sp2 bonds and made up of layers of mutually interconnected hexagonal rings. The layers are arranged in parallel planes 335 pm apart. Carbon atoms in the adjoining layers are not chemically bonded to each other and they are attached by weak vander Waals forces that make it possible for various atoms or molecules in liquid or gaseous form to get in between the carbon layers. The resulting substances are called intercalation compounds of graphite and their characteristic parameter is the so-called "degree of intercalation", which indicates the number of carbon layers between two layers of an intercalated substance [1].

Depending on a type of the intercalated substance the graphite plane may be either an acceptor or donor of electrons. Another option is the so-called π–complex created by intercalation of substances of AXy type, where A is a metal or non-metal with a high valence status, X is an electronegative element and y is a stoichiometric coefficient.

Intercalates of graphite with alkali metals have been known since 1930s. They are called intercalates of the first degree with the formula C_8M (M=K, Rb, Cs), i.e. they are characterized by a stacking sequence of layers of carbon and alkali metal.

Intercalates of graphite with alkali metals or in combination with other metals have been used in a number of applications as catalysts, e.g. for synthesis of ammonia, synthesis of carbohydrates by hydrogenation of carbon oxides, hydrogenation of olefins, they have sorption properties etc. [1]. Substituents can be chemically bonded to graphite under certain conditions by fluorination or oxidization.

Fluorination of graphite with elemental fluorine at 400-600°C produces a covalent compound called fluoro graphite CF_x, x = 0.25-1.12, depending on reaction conditions of the fluorination [1].

Oxidization of graphite with strong oxidizing agents (a combination of $KMnO_4$, $KClO_4$, $NaNO_3$ and H_2SO_4) produces graphene oxide (GO), which is a precursor for chemical preparation of graphene [2].

GO is a compound made up of a carbon skeleton with main functional groups, such as carboxyl, carbonyl, epoxy and ether groups and hydroxy groups. These functional groups enable chemical reactions of GO [3] to form covalent bonds with other compounds (e.g. esterification, amidation).

Another option is a GO reaction to form non-covalent bonds [2]. The possible types of the bonds are hydrogen bonds, van der Waals forces, H-π, cation-π, anion-π, π–π, electrostatic forces. These non-covalent bonds are employed in preparation of composite polymers, biopolymers [4] and in use of adsorption and absorption properties of GO [5-7]. GO suspension can be vacuum filtered to prepare foils that find use in biology, electrical engineering, optics [8] and biomedicine [9].

Graphene can be prepared by a chemical method which consists in reduction of oxidized carbon (functional groups) in GO with various reducing agents (hydrazine, metal hydride, hydrogen, hydrogen iodide) or reducing methods, such as reflux in a polar solvent, microwaves irradiation, electrochemical reduction [12].

Another carbon modification from the nano particles group are fullerene sand their best known representative is fullerene C_{60} with a spherical molecule. Fullerene molecule may under gomostlynucleophilic and radical reactions [10].

Fullerenes are condensed polycyclic carbon substances with a cage structure and with even numbers of carbon atoms arranged preferably into

Author α: VŠB-Technical University of Ostrava, Faculty of Safety Engineering, State Office for Nuclear Safety, Prague.
Author σ: State Office for Nuclear Safety, Prague.
e-mail: eva.zemanova@sujb.cz
Author ρ: Ministry of the Interior – General Directorate of the Fire Rescue Service of the Czech Republic, Technical Institute of Fire Protection.
Author ω: National Institute for Nuclear, Chemical and Biological Protection Kamenná.

pentagons or hexagons. The most perfect spherical shape has the fullerene C_{60}. Its surface is made up of twenty hexagons and twelve pentagons (similar to a classical sewn soccer ball). The pentagons are made of single covalent bonds and the hexagons are made up of a system of alternating single and double bonds.

The principal difference from graphite and diamond is the solubility of C_{60} in non-polar organic substances and its reactivity. Its typical reactions are associated with a transformation of arrangement from sp^2 to sp^3 and thus with a reduction of tension in the molecule. The C_{60} molecule is electropositive which means that it prefers nucleophilic or radical addition on a multiple bond. Fullerene C_{60} can be used for hydrogenation, alkylation, halogenation, oxidation, polymerization etc.

The contribution describesour "combination experiment"

- Bromination of fullerene C_{60} + sub sequentreaction with GO
- Bromination of GO-C_{60} foil and combination of GO with C_{60}

Functional groups of the resulting products were identified with FT-IR. We also determined their thermal stability which is the main topic of this contribution.

II. Experimental Part

- Employed chemicals

Graphite PM – very fine crystalline powder graphite, mesh 0.025mm.

Fullerene C_{60}, 99.5% purity, SES Research, Houston USA.

Sulfuric acid, nitric acid, potassium permanganate, pyridine, tetrahydrofuran, liquid bromine, Supplier: Sigma – Aldrich.

Ultrasonification with PS 400A, power output 500 W, thermostat 75°C, frequency 35 Hz.

a) Measuring Instruments

ATR analysis by means of FTIR spectrometry was performed using the spectrometer Brucker Aplha/FT-IR, ART crystal (identified as Platinum Diamond 1 Ref1), software OPUS 6, 5, source j IR SiC Globar. The number of spectrum scans was 24, resolution 4 cm-1, spectrum range 375-4000 cm-1.

Thermal analyses TGA and DSC of the prepared samples were performed on STA 1500, Instrument Specialists Incorporated-THASS, analytical scale SUMMIT, SI 234-4, at flow rate 20 ml/min, heating rate 10°C/min, ceramic crucible, diameter 5 mm and height 8 mm, degradation medium air. Morphology of the products was determined with SEM Phenom FEI and SEM FEI Quanta 650 FEG (USA).

b) Preparation of Graphene-Oxide (Hereinafter GO) by Oxidization of Graphite and Preparation of Graphene-Oxide-C_{60} (GO-C_{60}) by Oxidization of Graphite and C_{60} Mixture

i. *Sample weights for the individual experiments*

- 1 g graphite, 0.5 g C_{60}, 2.11 g NaNO3, 4.6 g KMnO4, 35 ml H2SO4
- 2 g graphite, 2.8 g NaNO3, 6.5 g KMnO4, 45 ml H2SO4
- Graphite and mixtures of graphite + C_{60} and GO+C60 were oxidized with a mixture of H_2SO_4, $KMnO_4$ and $NaNO_3$ according to Hummers and Offer man [11]. Graphite, sulfuric acid and sodiumnitrate (in the case of experiments I also fullereneC60) were placed into aflask, the mixture was stirred and cooled to10°C.

Potassium permanganate was subsequently added into the reaction mixture through a hopper in small doses. The mixture with the permanganate was slowly heated to 60°C and stirred at that temperature for 3 hours. Then it was left to stand for three days at the laboratory temperature.

The obtained products were filtered off, washed with a big quantity of distilled water until negative reaction to sulfate anions and dried for three days on a Petri dish at 50-60°C to form foils of GO and GO-C_{60}.

c) Bromination of Fullerene

FullereneC_{60} (4 g) was added into liquid bromine 27.5 ml (85.3 g) and the mixture was agitated at the laboratory temperature for 72 hours. The excessive bromine was removed by drying at 75°C for 24 hours until constant weight. The yield was 9.9 g of green-brown sub stance. According to the weight increase and subsequen telemental analysis, the average composition was $C_{60}Br_{14-18}$.

d) Modification of Graphene - Oxide (GO) by Reaction with Fullerene Bromo Derivative

GO (0.23 g) from the foil that was cut into tickets sized 2x5 mm was placed into THF (25 ml). The mixture was ultrasonificated for 10 minutes at the laboratory temperature to form suspension of GO in THF. Subsequently, we added fullerene bromo derivative (0.4 g) and 0.3 ml of pyridine. The suspension was brown. The reaction mixture was ultrasonificated for 10 minutes and then left for 24 hour sand intermittently stirred. The suspension was yellow-brown. The solid component was vacuumfiltered off and the filter cake was washed with 25 ml HCl (1:3) and 40 ml THF and subsequently dried at 50°Cfor 2 hours. The process produced 0.6 gof the product.

e) Bromination of GO-C_{60} with Liquid Bromine

The GO-C_{60} foil (0.15 g) was ultrasonificated in a flask in 10 ml of water solution and then liquidBr2 (4-5

ml) was added. There action mixture was left to stand for 15 day sat the laboratory temperature and intermittently stirred. The content of the flask was poured out on a Petri dish and gradually evaporated.

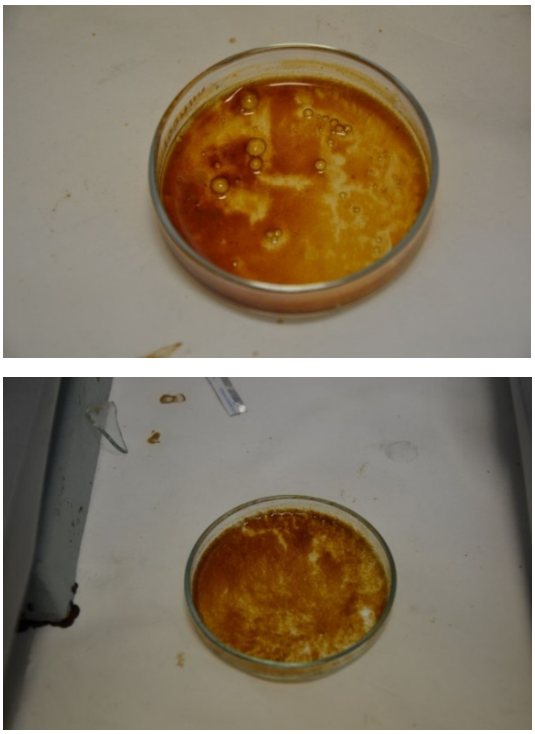
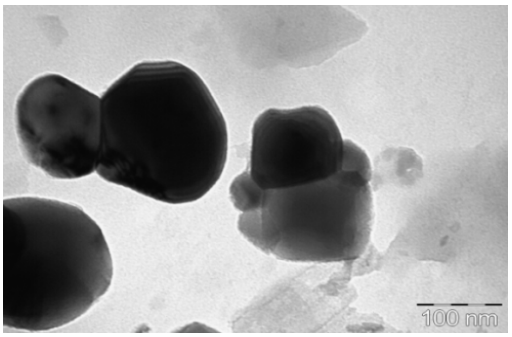


Fig. 1 : Gradual evaporation of brominated GO-C_{60} at 35-45°C; reaction of GO-C_{60} with Br Lin water environment

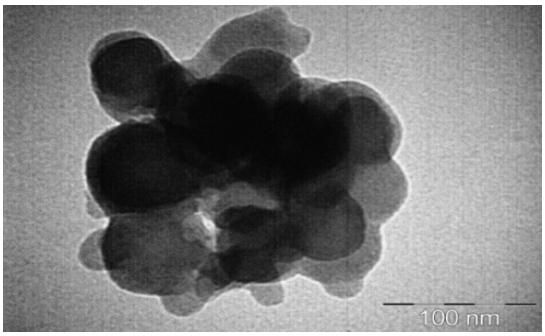
III. Results

a) Identification of Bromo fullerene

There is a number of publications relating to halogen derivatives of fullerenes [13-20] and describing preparation and identification of bromo and chloroderivatives of fullerenes C_{60}. The content of bromine in C_{60} Br_n is defined in the range2 < n < 24 depending on the reaction conditions (direct contact with bromine, reactionina solvent, reaction time and temperature, etc.), while the bromine content greater than n=24isascribed to an adduct with bromine; also adducts with a solvent have been described in those cases where the bromination is performed insolvents, e.g. in CS_2, $CHBr_3$, $C_6H_4C_{l2}$ etc. In our case the bromo derivative of fullerene was prepared by direct contact with liquid bromine. The average composition of the product was determined by elemental analysis and by surface analysisasC_{60} Br^{14-18}.



a) C_{60}



b) C_{60} Br_{14-18}

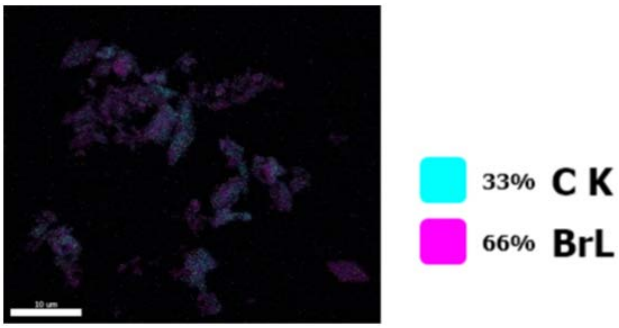


Fig. 2 : a) Electron image of fullerene, b) bromo fullerene c) Energy-dispersive X-ray Spectroscopy (EDAX)

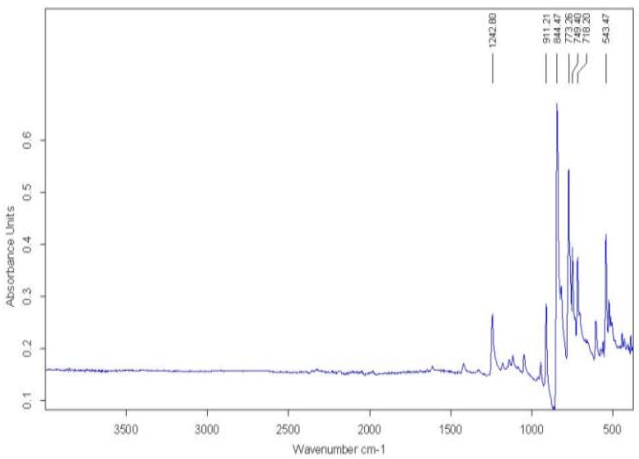


Fig. 3: IR spectrum of the bromo derivative of fullerene

FT-IR spectrums were used to identify the following vibrations: 1242 w, 911 w, 844 vs, 773 vs, 749 m, 718 m, 543 m (cm^{-1}) - Fig. 3,the strongest of which is844 cm^{-1} and it corresponds to the published data of the strongest vibrations for the bond C-Br. We have also provided the IR spectrum of the initial fullerene C_{60} for comparison (Fig. 4). $C_{60}Br_{24}$851 cm^{-1} [14], $C_{60}Br_8$849 cm^{-1} [14], C_{60} Br_8 847 cm^{-1} [15], $C_{60}Br_{24}$849 cm^{-1} [15], C_{60}Br14842 cm^{-1} /[15], C_{60} Br 8•2Br2 720 cm^{-1}, 846 cm^{1} [15], C_{60} Br_{14}•Br2 842 cm^{-1}[16], $C_{60}Br_{24}$•2xBr2 846 cm^{-1} [16].

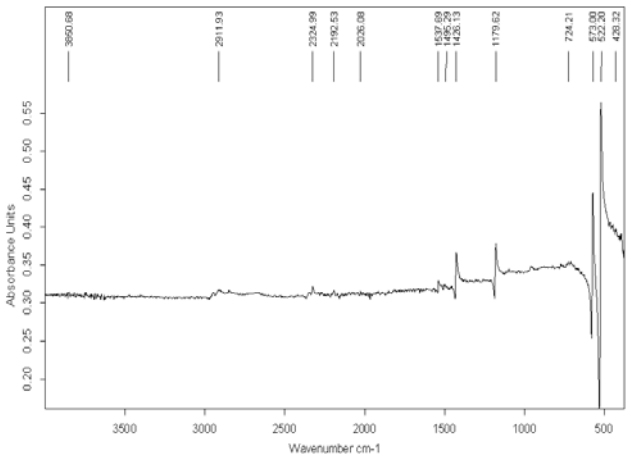


Fig. 4 : IR-spectrum of C_{60}

The following data were published on thermal stability of the prepared products of fullerene bromination:

$C_{60}Br_{24}$- 2 levels of decompositionat 45°C and170°C [16]

$C_{60}Br_{24}$- at100°C the beginning, the maximum at162°C [17-18]

$C_{60}Br_{24}$- at 90°C [14]

$C_{60}Br_8$- at 70°C–the beginning of decomposition [14]

The pressure of developed gasesre leased by decomposition of C_{60} Br_6 was measured [17] and based on the obtained values the authors assumed gradual decomposition with partial maximums at 138°C, 175°C and 204°C while in the temperature interval 90-187°C1 atom of bromine will bereleased from the cyclopentanecircle and cyclopentadienyl radical will operate as an inter media test age of the decomposition. Then 5 remaining atoms of bromine are expected to be released. The author santicipate a similar course of decomposition also for C_{60} Br_{24}.

For our C_{60} $Br_{14\text{-}18}$ the shape of the DSC curvesis different in the end other mic process area. In this area we anticipate release of bromine. For the bromo derivative alone the temperature range of the end other mic process is 62°C (126-188 °C) and the shape of the DSC curve is protracted(see Fig.No.5) which may hide partial maximum sand indicate a step-like release of bromine eviaintermediate products of decomposition, e.g.C_{60} Br_8.

The endothermic effect is 326.8 kJ/kg with the weight loss of 57%, the exothermic effect occurs in the temperature interval 418 – 504 with ΔH 4233 kJ/kg with the weight loss of 32%.A 10% weight loss of the sample occurred between the endothermic and exothermic processes (see Fig. 5).

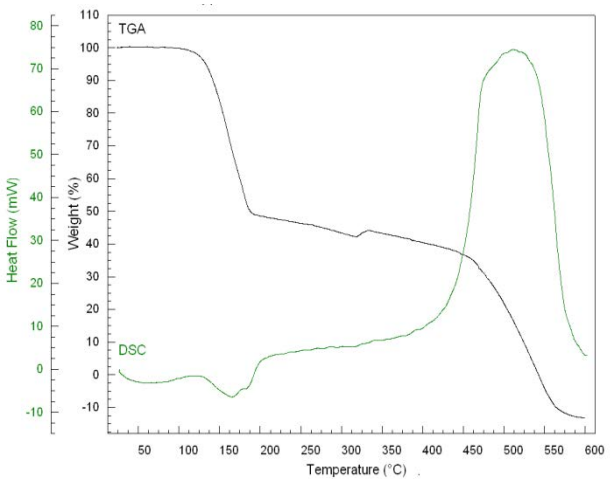


Fig. 5 : Thermal analysis of the initial bromo derivative C_{60} $Br_{14\text{-}18}$ (degradation medium: air, air flow rate 20 ml/min, temperature 25-600° C, heating rate 10°/min, sample weight 9.3 mg)

In order to confirm the main source of the weight loss during the end other mic process we heated the sample to 250°C and analyzedit with FT-IR spectroscopy. The obtained spectrumin Fig. 6 corresponds to the IR spectrum of the initial fullerene (compare Figures 4 and 6). This confirms our assumption that all bromine is released up to the temperature of 250°C.

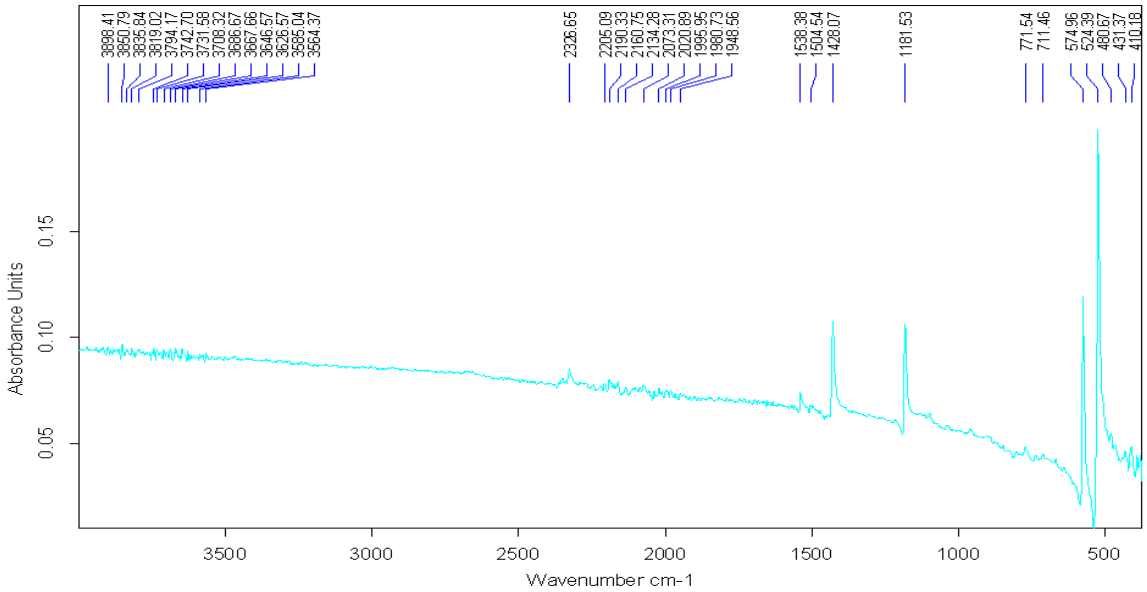


Fig. 6 : IR spectrum of fullerene bromo-derivative after heating to 250° C

b) Modification of Graphene-Oxide by Reaction with Fullerene Bromo Derivative

The measured spectrum of the obtained product is shown in Fig. 7. Spectrums of the initial substances are provided in Fig. 8 and Fig. 3 for comparison.

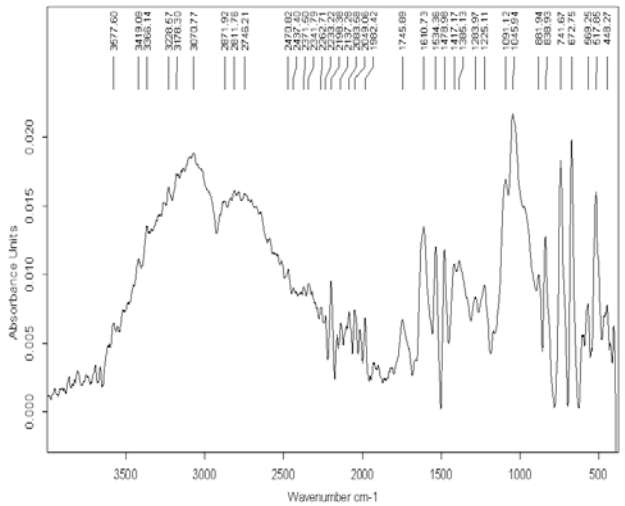


Fig. 7 : IR spectrum of a product of the reaction of GO with fullerene bromo derivative

Dominant vibrations:

For GO the skeletonvibration is at 1613 cm-^{1} and the vibration of C=C bonds in GO-C60Brisat 1610 cm-^{1} and it is no more dominant.

There are new dominant vibration sat 1091 cm-^{1} and 1045 cm-^{1} that include bond vibrations of a whole range of possibilities from epoxides, hydroxyles, C_O_C (for GO the value was 1068 cm_^{1}).

A similar range of the potential groups for GO is covered by vibrations at 1068 cm-1 and 979 cm-^{1}. Vibration scharacterizing the groups C=O for GOC_{60}Brx shifted towards the higher value 1745 cm-1in comparison to1727 cm-^{1} for GO.

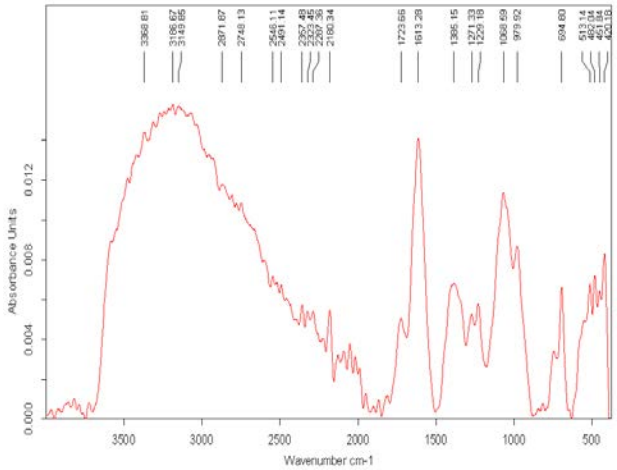


Fig. 8 : IR spectrum of the initial GO

New vibrations characterizing C-Br bonds were measured as dominant for GO C_{60} Brxat741 cm-1and 672 cm-^{1} (the value of 845 cm-^{1} for C-Br in the initial bromoderivative, which was dominant in it, was also found for the same bond in GO C_{60}Brx with a lower absorbance value – 839 cm-^{1}). The broad absorbance band 2400-3500 cm-^{1} was divided, which probably corresponds to the O-H valence vibration of new carboxyl functional groups. Newvibrations, which had not been identified for GO and bromo fullerene, appeared for the productat1534 cm-^{1}, 1478 cm-^{1} and 1417 cm-^{1}.

c) Thermal Tests of the Products

Fig 9.Thermal analysis of a product of GO reaction with fullerene bromo derivative (degradation

medium: air, air flow rate 20ml/min, temperature 25-600°C, heating rate 10°/min, sample weight 8.6 mg).

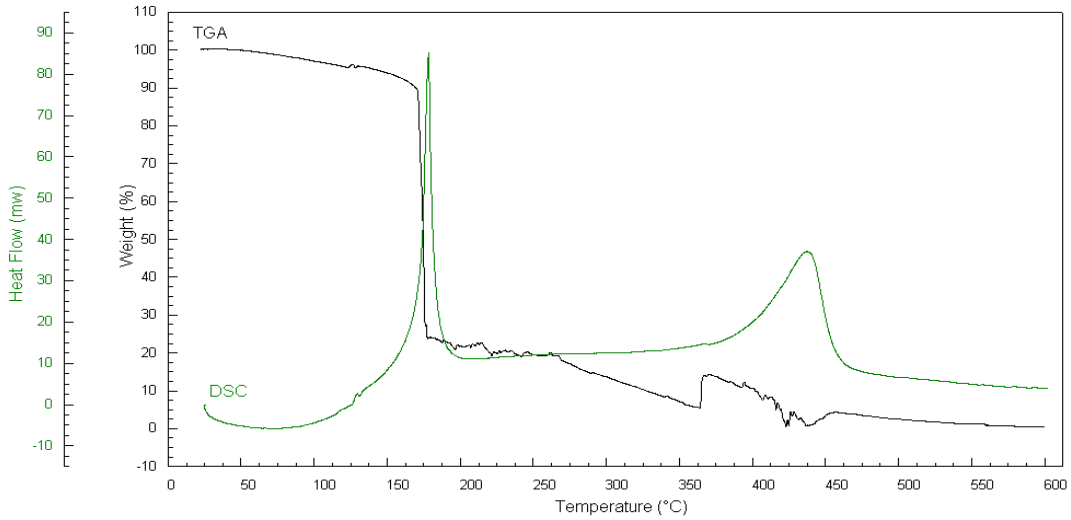


Fig. 10 : Thermal analysis of GO foil (degradation medium: air, air flow rate 20 ml/min‡emperature 25-600°C, heating rate 10°/min, sample weight 10.0 mg).

The comparison of thermal stabilities of the initial GO, the used fullerene bromo derivative and the prepared product has shown that the first exothermic process starts for GO at190.9°C with the maximum at 225°Cand with the thermal fect of 508.4 kJ/kg. For the prepared product the first exothermic process starts at atemperature by 40°Clower, with the maximum at 64°C and with a lower thermal effect, specifically 385.9kJ/kg– see Fig. 9,10andTab.1, 2). The thermal effect of the second exothermic process is also lower and it is approximately one half of that of GO. The weight loss during the first exothermic process of GO is more than 20% lower than that of the prepared product. In case of weight loss during the second process the situation is reverse. The total thermal effect of the decomposing reactionis significantly higher than in the case of theinitial GO. For the fullerene bromoderivative alone the decomposition (debromination) occurs in the temperature interval 124-184°C with an endothermic effect (161kJ/kg) and with a significant weight loss of 47.6%. This explains the lower thermal effect during the first exothermic process of the prepared product and the higher weight loss than for GO alone. Further decomposition of the initial fullerene bromo derivative occurs in the temperature interval 459-569°C, which is ca. by 60°C higher than the temperature of the second exothermic process of the prepared product.

Table 1 : Division of the TGA curve into temperature intervals

SampleNo.	Thermal processNo.	Temperature range (°C)	Weight loss (%)
GO-C_{60}Brx (x=14-18)	1	25.0 – 169.4	10.1
	2	169.4 – 178.0	66.3
	3	178.0 – 268.3	5.1
	4	268.3 – 362.0	13.0
	5	362.0 – 458.7	1.4
	6	458.7 – 600.0	3.8
GO foil	1	25.0 – 142.4	11.0
	2	142.4 – 213.5	8.8
	3	213.5 – 222.3	43.6
	4	222.3 – 368.8	2.8
	5	368.8 – 473.0	18.1
	6	473.0 – 600.0	6.1

Table 2 : Parameters of the ongoing thermal processes (DSC)

Sample No.	Thermal processNo.	Temperature range (°C)	ΔH (kJ/kg) *	H_{fi}(mW)	ΣΔH (kJ/kg)
GO-C_{60}Brx (x=14-18)	1	25.0 – 126.5	517.1	8.2	-517.1
	2	150.5 – 195.4	-385.9	75.5	
	3	366.9 – 465.8	-648.9	22.5	
GO foil	1	25.0 – 154.1	874.6	15.8	-910.6
	2	190.9 – 241.1	-508.4	107.4	
	3	356.5 – 492.1	-1277.1	31.0	

**ΔH = thermal effect of the process based on the DSC curves*
(ΔH > 0…endothermic process, ΔH < 0…exothermic process)

Fig 11.Shows the complex surface structure of the prepared product GO-C_{60} Brx (x=14-18).

d) Reaction of GO-C_{60} Foil with Liquid Bromine

i. *Evaluation of the IR Spectrum of GO-C_{60} Foil Brominated with Liquid Bromine*

The IR spectrums of GO-C_{60} and its brominated product (Fig. 12 and 13) demonstrate the following general differences: wave number shifts, changes in values of absorbances and changes in intensities of vibrations with common assignment for both the substances , such as e.g. changes of mutual intensities of vibrations of the –C=C– bond in respect to C-O-C (from 1:1 for GO-C_{60} to 1:3 for [GO-C60] Brx), increase in vibration intensities of the bonds –C=O or -COOH compared to –C=C- (3x for [GO-C_{60}]-Brx).

The main shift in the vibrations occurred for the broad band characterizing bond vibrations of the –OH group, where the maximum of absorbance for GO-C_{60} is at the value lower by ca. 200 cm-^{1}. The vibration of the C-O-C group for GO-C_{60} has the wave number higher by 24 cm-^{1}.

A significant difference of the IR spectrum of the brominated derivative is the presence of vibrations at 871 cm-^{1} and 571 cm-^{1}, to which we have assigned the valence vibration of the C-Br bond, and the presence of a new strong vibration at 1153 cm-^{1}, which can be assigned both to the deformation vibration of the C-Br bond and to deformation of C-CO-C and valence of C-O. Another difference consists in the fact that the spectrum of the bromo-product practically lacks any absorbance in the interval 1220-1380 cm-^{1}, which had been present as medium strong in the spectrum of the initial GO-C_{60} and which we had assigned to the epoxy groups, deformation of –OH and etheric groups.

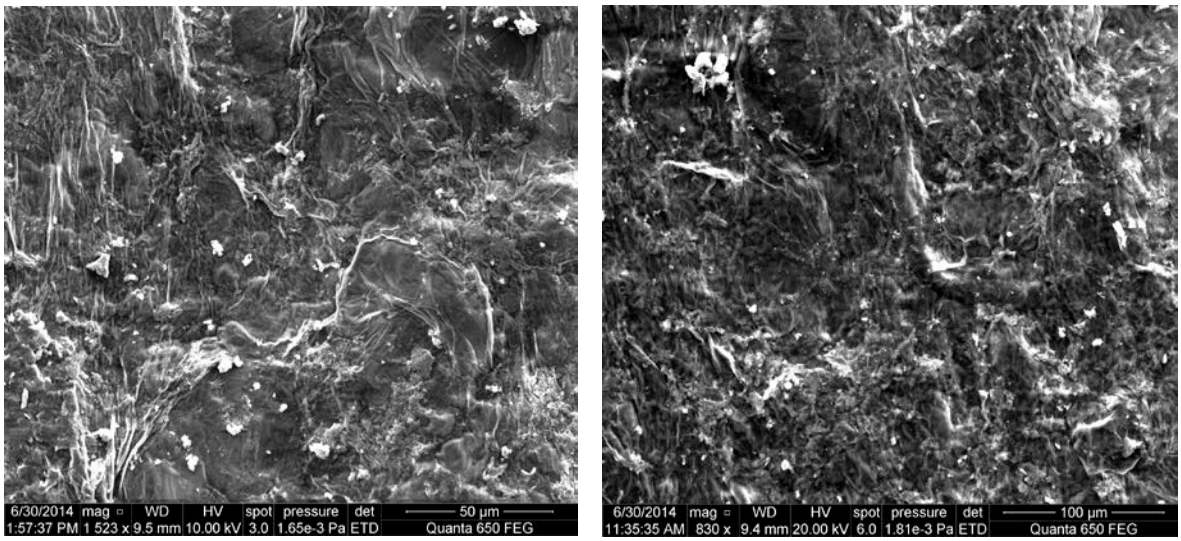


Fig .11 : Electron image of a product of GO reactio n with $C_{60}Br_{14-18}$, different scales

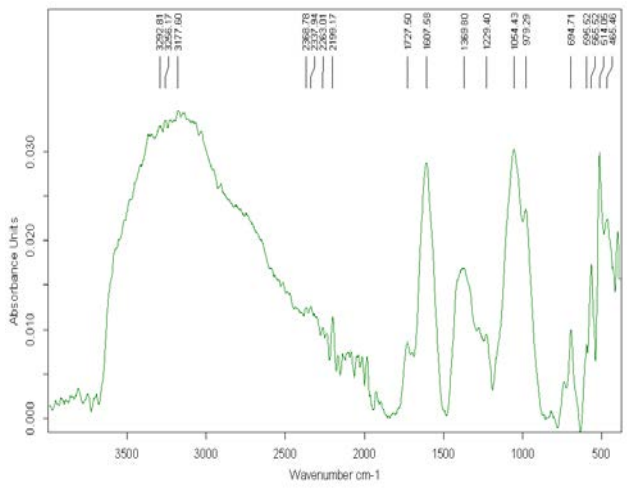


Fig. 12 : IR-spectrum of the initial GO-C_{60}

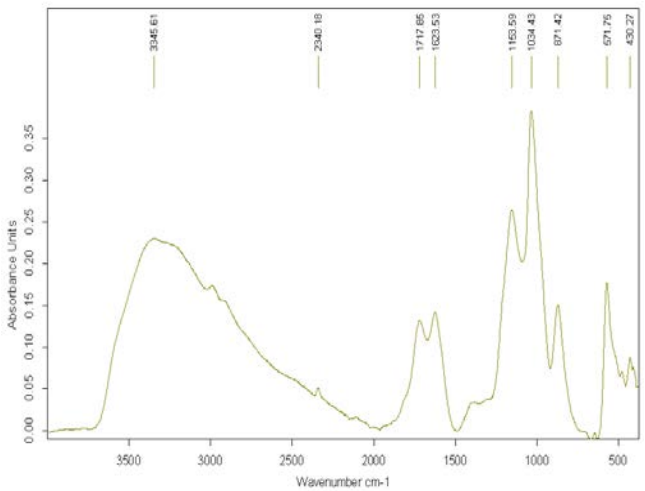


Fig. 13 : IR-spectrum of [GO- C_{60}]-Brx

e) Thermal Stability of the GO-C_{60} Foil Brominated with Liquid Bromine

For the brominated GO-C_{60} productthe thermal analysis has demonstrate done endothermic process and two exothermic processes(Fig. 14).Unlike the TGA analysis of the GO-C_{60} foil, the brominated product $C_{60}Br_{14-18}$,when thermally exposed, does not manifest such a sharp weight loss as the GO-C_{60} foil. In the temperature interval 213 - 222°Cthe weight loss was 44% (Fig. 15) and forC60Br14-18in the temperature interval 126 – 188°Cthe weight loss was 56% (Fig.5).Therefore, with a certain approximation, we can assume a gradual loss of weight (Tab. 3).The endothermic process in the product brominated with liquid bromine comes earlier than in the case of C_{60}Br14-18andits thermal effect is ca. 4 times bigger. The second exothermic process starts at a higher temperature in comparison with GO-C_{60}, while the first exothermic process occurs at a lower temperature.

Also interesting is the overall result of the thermal processes in the course of thermal decomposition, which is significantly smaller for brominatedGO-C60foil (301kJ/kg) than for the initial GO-C60foil (1204 kJ/kg). For C_{60} Br_{14-18} it is up to 3906 kJ/kg. In this case an important role in the total thermal effect is probably played by the considerable size of the endothermic effect of the brominated foil.

Bromine water with oxidizing effects may cause an increase in the number of carbonylorcarboxyl groups, due to splitting of the C-O-C bond. This can be concluded from the IR spectrum (absence of vibration sin the interval 1220-1380 cm^{-1}). Moreover, it is not possible to exclude addition of-OH Br+ on the moleculeor substitution of H+ with bromine in the carboxyl. Based on a comparison with the size of the endothermic effect in $C_{60}Br_{14-18}$ we anticipate various types of bromine bonds to the molecule. The size of the endothermic effect may also result in formation of anadduct of bromine and the molecule.

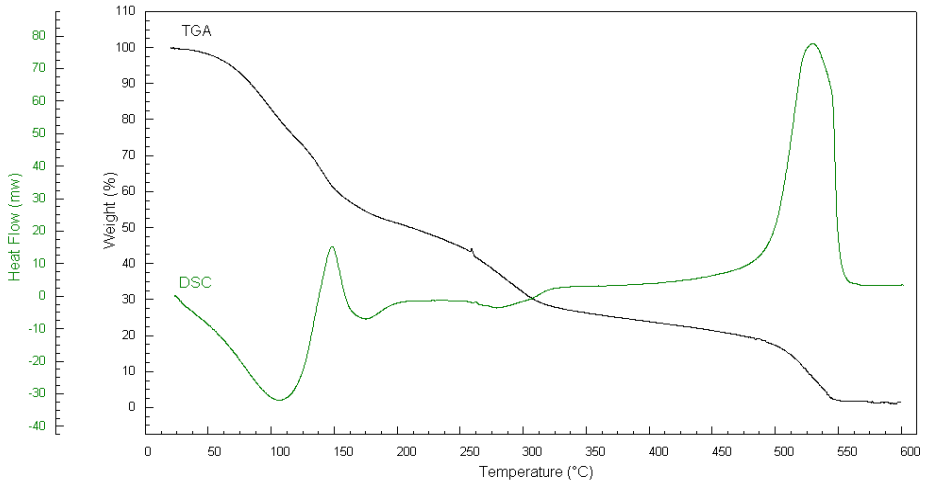


Fig. 14 : Thermal analysis [GO- C_{60}]-Br (degradation medium: air, air flow rate 20 ml/min, temperature 25-600°C, heating rate 10°/min, sample weight 10.05 mg)

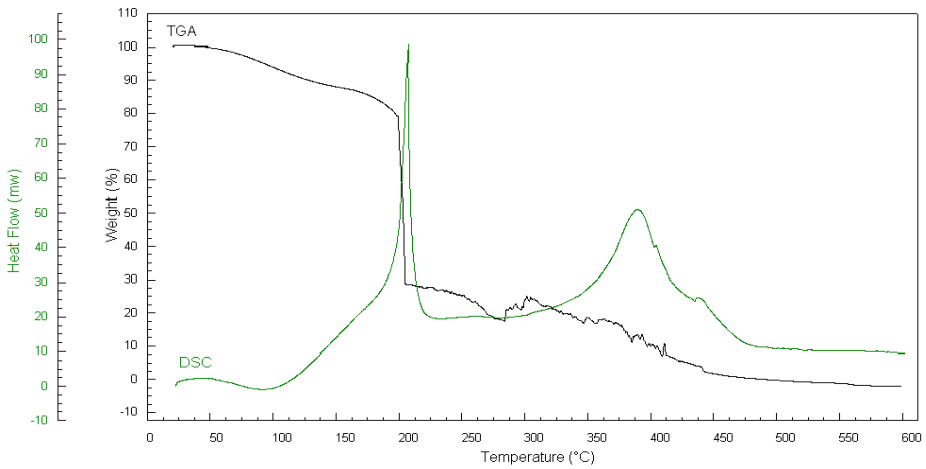


Fig. 15 : Thermal analysis foil GO-C_{60} (degradation medium: air, air flow rate 20ml/min, temperature 25-600°C, heating rate 10°/min, sample weight 10.0mg)

Table 3 : Division of the TGA curve into temperature intervals x

SampleNo.	Interval No.	Temperature range (°C)	Weight loss (%)
GO-C_{60}-Br	1	25.0 – 56.7	2.7
	2	56.7 – 114.0	20.3
	3	114.0 – 165.1	20.3
	4	165.1 – 259.4	12.6
	5	259.4 – 313.1	14.8
	6	313.1 – 504.1	12.6
	7	504.1 – 546.6	14.3
	8	546.6 – 600.0	0.9
GO-C_{60}foil	1	25.0 – 87.1	4.4
	2	87.1 – 153.0	8.2
	3	153.0 – 197.0	8.0
	4	197.0 – 205.0	51.1
	5	205.0 – 281.3	10.8
	6	281.3 – 490.9	18.0

X the indicated intersections of tangents to the respective bends of the TGA curve

Table 4 : Parameters of the ongoing thermal processes (DSC)

SampleNo.	Thermal effect No.	Temperature range(°C)	ΔH (kJ/kg) *	H_{fl} (mW)	ΣΔH (kj/kg)
GO-C_{60}-Br	1	25.0 – 131.9	1288.6	32.0	
	2	131.9 – 171.9	-180.1	21.8	-301.3
	3	253.3 – 322.8	81.5	2.0	
	4	478.6 – 557.3	-1490.7	67.2	
GO-C_{60}foil	1	42.0 – 124.2	141.7	6.4	
	2	182.6 – 221.5	-308.7	71.1	-1204.1
	3	319.7 – 481.6	-1037.1	28.1	

ΔH = thermal effect of the process based on DSC curves (ΔH > 0…endothermic process, ΔH < 0…exothermic process)
H_{fl} = height of the peak of a thermal process on the DSC curve in an absolute value related to the point corresponding to the beginning of the thermal process

IV. Discussion

Tens and hundreds of products (substances) have been described which were prepared by functionalization of graphene or graphene-oxide while forming both covalent and non-covalent bonds [21]. These products have different physicochemical properties. In the case of grapheme the reactions proceed via areactiveinter mediate, such as radical, carbene, aryne, nitreneornewly bonded substances [22]. Functional groups, such as–OH, -COOH, epoxy are used for the reaction of graphene-oxide. A classical example is a reaction with amines [23] when the group –COOH converts into COCl and the latter reacts readily with an amino group of the new substituent.

Graphene-oxide can be also functionalized with non-covalent bonds (van der Waals forces, hydrogen bonds, π-π interaction). As an example, we can provide its reaction with polymers, surface active substances, biomolecules etc. [24].

In the case of grapheme and fullerene a product has been described with anointer connection basedon π-π interaction. The product has interesting thermoelectric properties [25]. The interconnection of fullerene and GO with a covalent bond has been also described, specifically there action of substituted fullerene (-OH, -NH_2), fullerene pyrolidine, 1, 2 methano-fullerene– 61 – carboxyl acid [26-28] with active GO groups.

V. Conclusion

A direct reaction with liquid bromine was used to prepare bromo fullerene $C_{60}Br_{14-18}$. Further, graphene-oxide (GO) was prepared and reacted with the brominated fullerene. Results of the performed analyses have not positively shown whether the reaction of GO with $C_{60}Br_{14-18}$ lead to functionalization of GO with covalentor non-covalent bonds. Weassume potential partialesterification, also thanks to the alkalineenvironment. The shape of the thermal curve of a product of GO reaction with C_{60}Br14-18 is similar to that of GO and GO-C_{60}.

Another alternative was direct bromination of a GO-C_{60} mixture or foil created from the product. It was accompanied by partial oxidization, i.e. oxidization splitting of C-O-C bonds, epoxide bonds and bromination. The thermal decomposition of the product proceeded without external weight losses (unlike in the case of GO and initial GO-C_{60}).

In the process of thermal decomposition the prepared brominated GO and GO-C_{60} demonstrated lower thermal effects, which was caused by retardantability of bromine.

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Modeling, Simulation and Control of 2-R Robot

By Aalim M. Mustafa & A AL-SAIF
King Fahd University of Petroleum and Minerals, Saudi Arabia

Abstract- This article presents a study of Three PID controller technique of a 2-Revelutejoint robot. First we present Denavit-Hartenberg parameters for 2-R robot. Then we studied the dynamics of the 2-R robot and derived the nonlinear equations of motion. A PID controller has been implemented for three types of modeling technique: model based on linearization about equilibrium point, model based on Autodesk Inventor and Matlab/Simulink software's, and lastly model based on feedback linearization of the robot. A comparison between the three controllers is presented showing the effectiveness of each technique.

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GJRE-H Classification : *FOR Code: 090602p*

Strictly as per the compliance and regulations of:

Modeling, Simulation and Control of 2-R Robot

Aalim M. Mustafa [α] & A AL-SAIF [σ]

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I. Introduction

Robotics is the science that deals with robot's design, modeling and controlling. Nowadays robots are used everywhere in everyday life. It has accompanied people in most of industry and daily life jobs. (Gouasmi, Ouali, Fernini, & Meghatria, 2012).

The range of robot utilization is very wide. A large family of robots is used in industry and manufacturing. Robots are used in supplying the motion required in manufacturing processes such as pick and place, assembly, painting, milling, cutting, welding, drilling, etc.

Because of different types of tasks different manipulator configurations are available such as rectangular, cylindrical, spherical, revolute and horizontal jointed (Gouasmi et al., 2012).

A two revolute joint robot configuration with two degrees of freedom is generally well-suited for small parts insertion and assembly, like electronic components. Although the final goal is to design and manufacture real robotics, it is very useful to perform simulations prior to investigations with real robots. Simulations are easier to setup, less expensive, faster and more convenient to use. it allows better design exploration and helps you enhance your final real robot by selecting suitable parameters for the system you want to design (Žlajpah, 2008).

There are many control techniques used to control a robot arm. The most used ones are the PID control, optimal control, adaptive control and robust control. "There are many kinds of controllers that can be used to cause a designed robot arm to move along a desired trajectory" (Sukvichai, 2008). The simplest which we used in this paper to control the robot arm is the PID controller.

Author α: Mechanical Engineering department at King Fahd University of Petroleum and Minerals (KFUPM) Saudi Arabia.
e-mail: Aalim.motasim@hotmail.com
Author σ: System Engineering department at King Fahd University of Petroleum and Minerals (KFUPM) Saudi Arabia.

II. Problem Formulation

a) Robot Specifications

Consider the two joint sticks robot shown in figure (1) with the following specifications in Oxy coordinates:

L_1 = 1 m is the length of link 1

L_2= 1 m is the length link 2

m_1 = 1 kg is the mass of link 1

m_2 = 1 kg is the mass of link 2

θ_1Is the rotation angle of joint 1

θ_2Is the rotation angle of joint 2

L_{c1} =L_{c2}= 0.5 m is the distance to the half of the link.

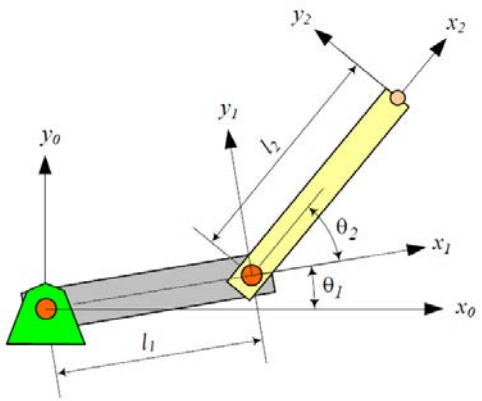

Fig. 1 : Two- joint 2-R Robot (N.Jazar, 2010)

b) Robot Kinematics

If we assigned the joints axes based on the Denavit Hartenberg representation, The (D-H) parameters for the 2-R robot will be defined as in the table below.

Table 1 : D-H parameters of 2-R Robot

Frame No.	a_i	α_i	d_i	θ_i
1	L_1	0	0	θ_1
2	L_2	0	0	θ_2

The initial position (at t = 0) from the homogeneous transformation matrix where $\theta_1 = 0^\circ \theta_2 = 0^\circ$ are shown in figure (2).

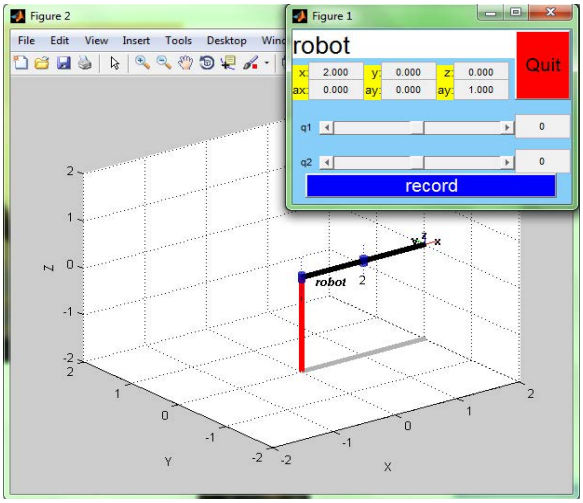

Fig. 2 : Home position of 2-R Robot

III. Robot Dynamics

Description of x and y in terms of θ_1 and θ_2 in term of linear displacement:

$x_1 = L_1 \sin\theta_1$

$y_1 = L_1 \cos\theta_1$

$x_2 = L_1 \sin\theta_1 + L_2 \sin(\theta_1 + \theta_2)$

$y_2 = L_1 \cos\theta_1 + L_2 \cos(\theta_1 + \theta_2)$

So, Kinetic Energy could be formed as:

$$KE = \frac{1}{2} m_1 v_1^2 + \frac{1}{2} m_2 v_2^2 + \frac{1}{2} j_1 \omega_1^2 + \frac{1}{2} j_2 \omega_2^2 \quad (1)$$

Substitute for v1 and v2

$$KE \frac{1}{2} m_1 l_{g1}^2 \dot{\theta}_1 + \frac{1}{2} m_2 \left(l_1^2 \dot{\theta}_1 + 2 l_1 l_{g2} \dot{\theta}_1 (\dot{\theta}_1 + \dot{\theta}_2) \cos\theta_2 + l_{g2}^2 (\dot{\theta}_1 + \dot{\theta}_2)^2 \right) + \frac{1}{2} j_1 \dot{\theta}_1 + \frac{1}{2} j_1 (\dot{\theta}_1 + \dot{\theta}_2)^2 \quad (2)$$

And Potential Energy is

$$PE = m_1 g l_{g1} \sin\theta_1 + m_2 g(l_1 \sin\theta_1 + (l_{g2} \sin(\theta_1 + \theta_1)) \quad (3)$$

a) Equations of motion

The Lagrangian of a dynamic system is defined as the difference between the kinetic and potential energy at an arbitrary instant (N.Jazar, 2010).

$$L = KE - PE$$

So, by Lagrange Dynamics, we form the Lagrangian

$$\mathcal{L} = l_{g1}^2 \dot{\theta}_1 + \frac{1}{2} m_2 \left(l_1^2 \dot{\theta}_1 + 2 l_1 l_{g2} \dot{\theta}_1 (\dot{\theta}_1 + \dot{\theta}_2) \cos\theta_2 + l_{g2}^2 (\dot{\theta}_1 + \dot{\theta}_2)^2 \right) + \frac{1}{2} j_1 \dot{\theta}_1 + \frac{1}{2} j_2 (\dot{\theta}_1 + \dot{\theta}_2)^2 - m_1 g l_{g1} \sin\theta_1 - m_2 g(l_1 \sin\theta_1 - (l_{g2} \sin(\theta_1 + \theta_2)) \quad (4)$$

Using Lagrange to form generalized equations of motion in matrix form as:

$$\begin{bmatrix} m_1 l_{g1}^2 + m_2 l_1^2 + j_1 & m_2 l_1 l_{g2} \cos(\theta_1 - \theta_2) \\ m_2 l_1 l_{g2} \cos(\theta_1 - \theta_2) & m_2 l_{g2}^2 + j_2 \end{bmatrix} \begin{bmatrix} \ddot{\theta}_1 \\ \ddot{\theta}_2 \end{bmatrix} - \left(m_2 l_1 l_{g2} g \sin(\theta_1 - \theta_2) \right) \begin{bmatrix} \dot{\theta}_1 \\ \dot{\theta}_2 \end{bmatrix} + \begin{bmatrix} (m_1 l_{g1} + m_2 l_1) g \cos\theta_1 \\ m_2 l_{g2} g \cos\theta_2 \end{bmatrix} = \begin{bmatrix} M_1 \\ M_2 \end{bmatrix} \quad (5)$$

And the general form is:

$$H(\ddot{q}) + C(\dot{q}, q) + g(q) = M$$

IV. PID Controller based on Linear Model

We define new variables in order to convert the 2-R robot to an equivalent linear model.

$$x_1 = \theta_1 x_2 = \theta_2 x_3 = \dot{\theta}_1 x_4 = \dot{\theta}_2$$

$$\dot{x}_1 = \dot{\theta}_1 = x_3 \dot{x}_2 = \dot{\theta}_2 = x_4 \dot{x}_3 = \ddot{\theta}_1 \dot{x}_4 = \ddot{\theta}_2$$

Rewrite the equation of motion using these variables, and use new constants c_1 to $c_6$function of robot specifications to make equations in simple form

$$\dot{x}_4 = \frac{M_2}{c_5} - \frac{c_2 M_2}{c_5} \cos(x_1 - x_2) + \frac{c_3}{c_5} \sin(x_1 - x_2) x_4 - \frac{c_6}{c_5} \cos x_2 \quad (6)$$

$$\left[c_1 - \frac{M_2}{c_5} \cos^2(x_1 - x_2) \right] \dot{x}_3 = M_1 - \frac{c_2 M_2}{c_5} \cos(x_1 - x_2) - \frac{c_2 c_3}{c_5} \cos(x_1 - x_2) \sin(x_1 - x_2) x_4 + \frac{c_2 c_6}{c_5} \cos(x_1 - x_2) \cos x_3 - c_4 \cos x_1 \quad (7)$$

$$\dot{x}_1 = x_3 \quad (8)$$

$$\dot{x}_2 = x_4 \quad (9)$$

Now we can write the state-space model using linearization about the equilibrium point:

$$\theta_1 = -\frac{\pi}{2} \quad \dot{\theta}_1 = 0 \theta_2 = -\frac{\pi}{2} \dot{\theta}_2 = 0 \quad \text{M1= 0}$$
$$\text{M2= 0}$$

We Perform Taylor series expansion of the nonlinear functions and neglect high-order terms, to get the linearized model. At equilibrium point:

Linearization of the variable x_1 with respect to other variables:

$$\frac{\partial \dot{x_1}}{\partial x_1} = 0 \quad \frac{\partial \dot{x_1}}{\partial x_2} = 0 \quad \frac{\partial \dot{x_1}}{\partial x_3} = 1 \quad \frac{\partial \dot{x_1}}{\partial x_4} = 0$$

Linearization of the variable x_1 with respect to other variables:

$$\frac{\partial \dot{x_2}}{\partial x_1} = 0 \quad \frac{\partial \dot{x_2}}{\partial x_2} = 0 \quad \frac{\partial \dot{x_2}}{\partial x_3} = 0 \quad \frac{\partial \dot{x_2}}{\partial x_4} = 1$$

Linearization of the variable x_1 with respect to other variables:

$$\frac{\partial \dot{x_3}}{\partial x_1} = \frac{c_4 c_5}{c_1 c_5 - M_2} \quad \frac{\partial \dot{x_3}}{\partial x_2} = \frac{c_2 c_6}{c_1 c_5 - M_2} \quad \frac{\partial \dot{x_3}}{\partial x_3} = 0 \quad \frac{\partial \dot{x_3}}{\partial x_4} = 0$$

Linearization of the variable x_1 with respect to other variables:

$$\frac{\partial \dot{x_4}}{\partial x_2} = \frac{-c_6}{c_5} \frac{\partial \dot{x_4}}{\partial x_3} = 0$$

$$\frac{\partial \dot{x_4}}{\partial x_4} = \frac{c_3}{c_5} \sin(x_1 - x_2) \frac{\partial \dot{x_4}}{\partial x_4} = 0$$

Linearization of the variable x_1 and x_2 with respect to input torques:

$$\frac{\partial \dot{x_1}}{\partial M_1} = 0 \quad \frac{\partial \dot{x_1}}{\partial M_2} = 0 \quad \frac{\partial \dot{x_2}}{\partial M_1} = 0 \quad \frac{\partial \dot{x_2}}{\partial M_2} = 0$$

$$\frac{\partial \dot{x_3}}{\partial M_1} = \frac{c_5}{c_1 c_5 - M_2} \frac{\partial \dot{x_3}}{\partial M_2} = \frac{-c_2}{c_1 c_5 - M_2} \frac{\partial \dot{x_4}}{\partial M_1}$$

$$= 0 \quad \frac{\partial \dot{x_4}}{\partial M_2} = \frac{1 - c_2}{c_5}$$

We can write the state-space model:

$$\begin{bmatrix} \Delta \dot{x_1} \\ \Delta \dot{x_2} \\ \Delta \dot{x_3} \\ \Delta \dot{x_4} \end{bmatrix} = \begin{bmatrix} 0 & 0 & 10 \\ 0 & 0 & 01 \\ \frac{c_4 c_5}{c_1 c_5 - M_2} & \frac{c_2 c_6}{c_1 c_5 - M_2} & 00 \\ 0 & \frac{-c_6}{c_5} & 00 \end{bmatrix} \begin{bmatrix} \Delta x_1 \\ \Delta x_2 \\ \Delta x_3 \\ \Delta x_4 \end{bmatrix}$$

$$+ \begin{bmatrix} 0 & 0 \\ 0 & 0 \\ \frac{c_5}{c_1 c_5 - M_2} & \frac{-c_2}{c_1 c_5 - M_2} \\ 0 & \frac{1 - c_2}{c_5} \end{bmatrix} \begin{bmatrix} \Delta M_1 \\ \Delta M_2 \end{bmatrix}$$

$$Y = \begin{bmatrix} 1000 \\ 0100 \\ 0010 \\ 0001 \end{bmatrix} \begin{bmatrix} \Delta x_1 \\ \Delta x_2 \\ \Delta x_3 \\ \Delta x_4 \end{bmatrix} + [0][D]$$

a) Linearized model

We substitute values of constants c_1 to c_6 into the state-space model to get the state space matrices:

$$A = \begin{bmatrix} 0 & 0 & 10 \\ 0 & 0 & 01 \\ -0.4568 & -0.619600 \\ 0.2485 & \_6.617400 \end{bmatrix}$$

$$B = \begin{bmatrix} 0 & 0 \\ 0 & 0 \\ 0.7870 & -0.0426 \\ 0.0426 & 0.1349 \end{bmatrix}$$

$$[C] = \begin{bmatrix} 1 & 00 & 0 \\ 0 & 10 & 0 \end{bmatrix}$$

$$[D] = \begin{bmatrix} 0 & 0 \\ 0 & 0 \end{bmatrix}$$

b) Controller for Linear model

Applying state space matrices on Matlab Simulink, we formulate a model which can be controlled easily using the block (PID) in Simulink library. Figure (3) shows the linear model in Simulink. If we run the model we get the results shown in figure (4) and figure (5) for the angle θ_1 and θ_2 respectively. The input is step function with angle 45° for both links.

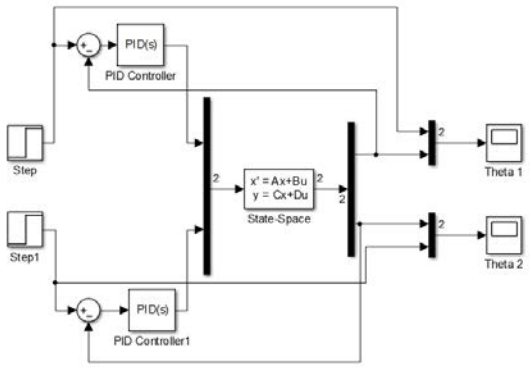

Fig. 3 : Simulink diagram for linearized model

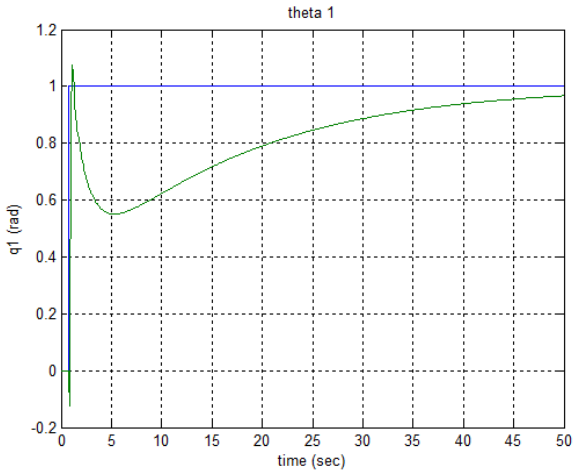

Fig. 4 : Dynamic response of link_1 (Linearized model)

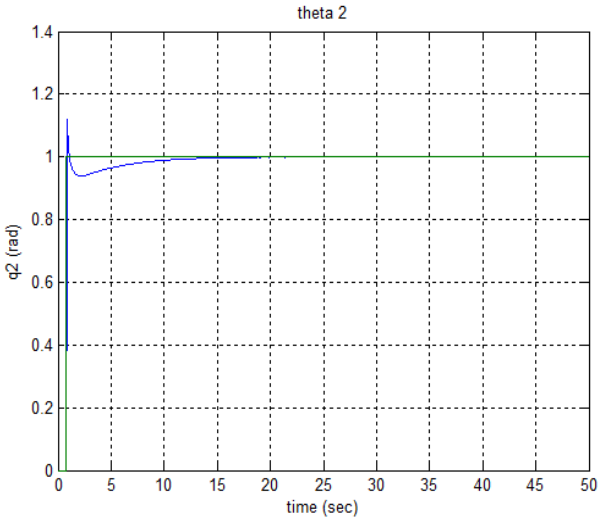

Fig. 5 : Dynamic response of link_2 (Linearized model)

It is clearly seen that the first link which have much inertia takes longer time to follow the desired trajectory. And both links have a delay in response.

V. PID Controller based on Autodesk Inventor Model

A 2-R robot system is designed and developed using Autodesk Inventor program and MATLAB/Simulink simultaneously as shown in Figure (6) and Figure (7).Robot specifications is taken into account while modeling. After that we transform the designed model into Simulink environment and automatically block diagram has been developed for the robot.

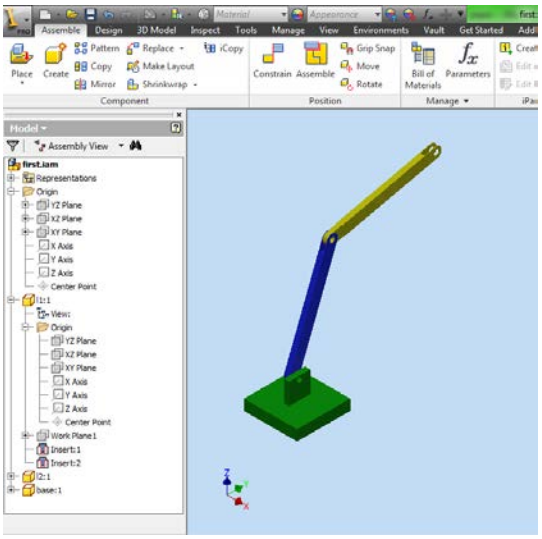

Fig. 6 : 2-R Robot in Autodesk Inventor

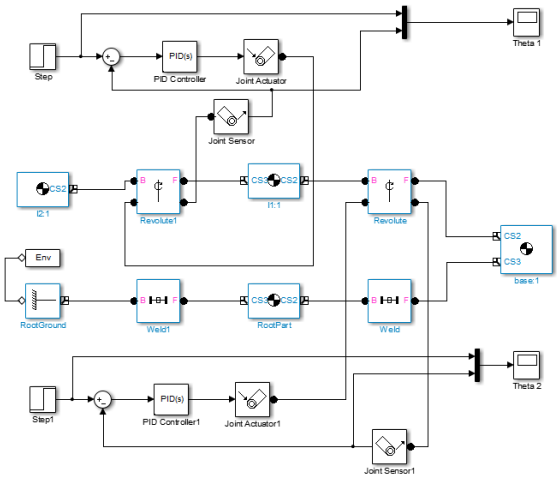

Fig. 7 : Simulink diagram for Autodesk Inventor model

a) Controller for Autodesk Inventor model

Applying a PID controller using the block (PID) in Simulink library we can control our system as shown in Figure (7) below. The results show much better response than the linearized model used in pervious part.

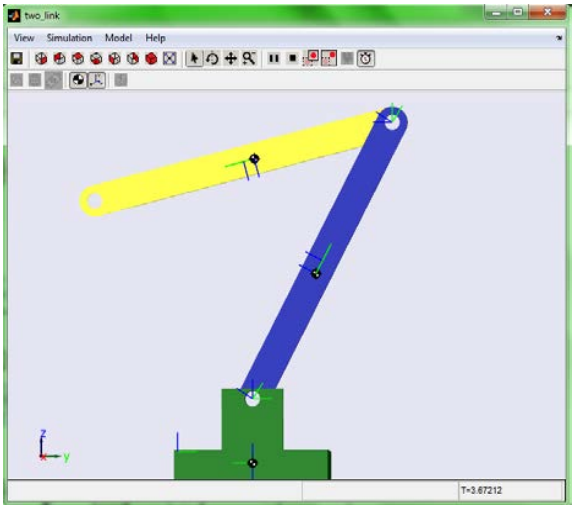

Fig. 8 : 2-R Robot simulation in Simulink

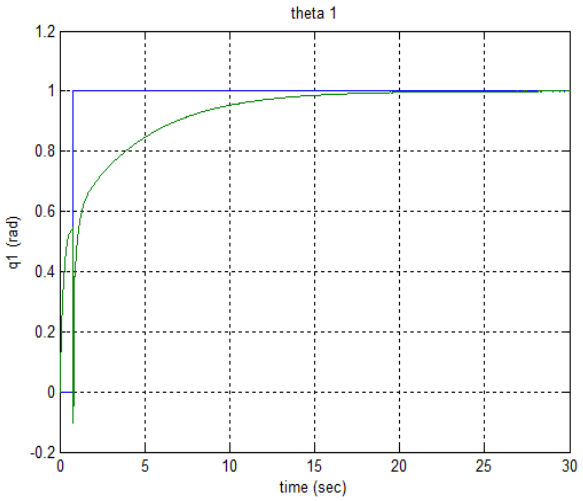

Fig. 9 : Dynamic response of link_1 (Autodesk Inventor)

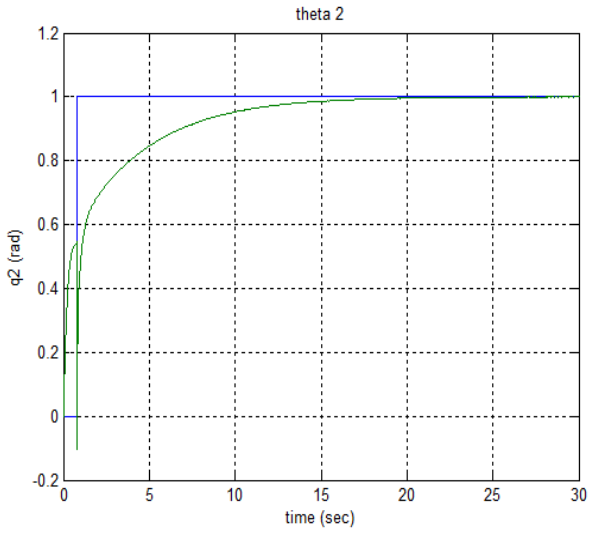

Fig. 10 : Dynamic response of link_2 (Autodesk Inventor)

VI. PID Controller based on Feedback Linearization

Having system's equation

$$H(\ddot{q}) + C(\dot{q}, q) + g(q) = M$$

$$\ddot{q} = H^{-1}[-C(\dot{q}, q) - g(q)] + \bar{M}$$

While: $\bar{M} = H^{-1} M$ And, $M = H^{-1}\bar{M}$

This way, we decoupled the system to have the (non-physical) torque input:

$$\bar{M} = H^{-1}\begin{bmatrix} M_1 \\ M_2 \end{bmatrix}$$

However, the physical torque inputs to the system are:

$$M = H\begin{bmatrix} \bar{M}_1 \\ \bar{M}_2 \end{bmatrix}$$

To design the feedback PID controller, error signals are assumed to be:

$$e\theta_1 = \theta_{1f} - \theta_1 e\theta_2 = \theta_{2f} - \theta_2$$

Assuming the final position desired is $\theta_{1f} = \frac{pi}{4}$, $\theta_{2f} = \frac{pi}{4}$ And the initial condition for the system is$\theta_{10} = 0 \quad \theta_{20} = 0$ General structure of PID controller for any input would be:

$$M = K_P e + K_D \dot{e} + K_I \int e\, dt$$

$$= K_{P1}(\theta_{1f} - \theta_1) + K_{D1}\dot{\theta_1} + K_{I1}\int(\theta_{1f} - \theta_1)\, dt \quad (10)$$

$$M_2 = K_{P2}(\theta_{2f} - \theta_2) + K_{D2}\dot{\theta_2} + K_{I2}\int(\theta_{2f} - \theta_2)\, dt \quad (11)$$

Applying equations (10) and (11) we got results shown below in figures (11) to (14).

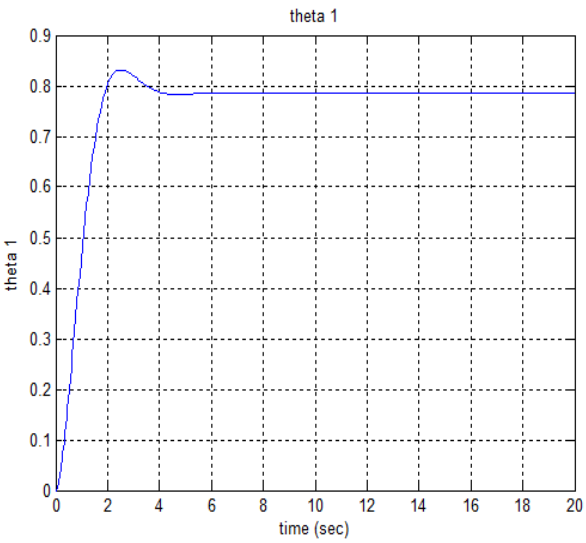

Fig. 11 : Dynamic response of link_1 (Nonlinear model)

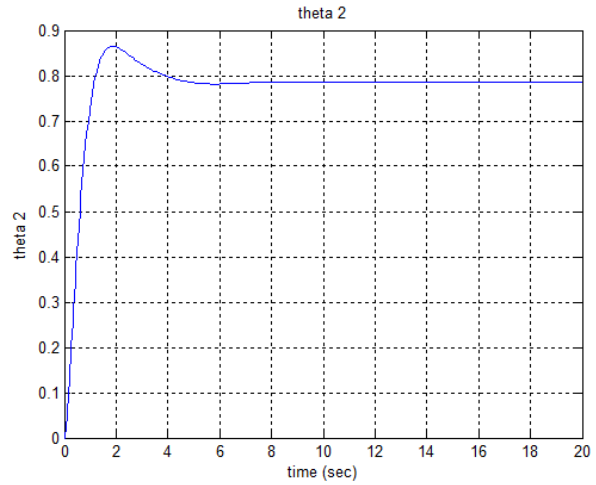

Fig. 12 : Dynamic response of link_2 (Nonlinear model)

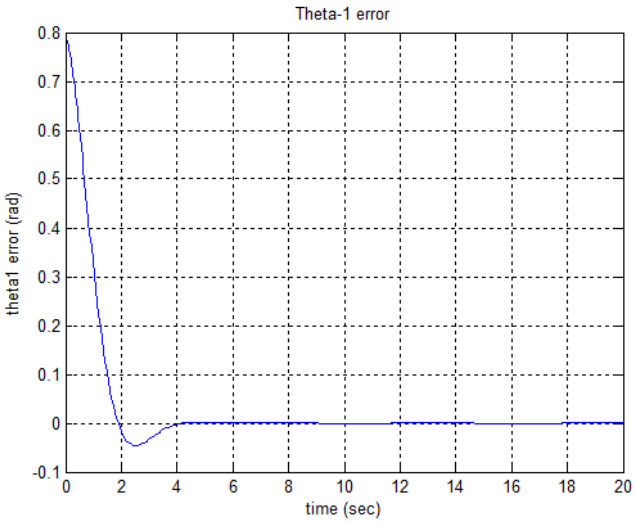

Fig. 13 : Error of theta_1 (Nonlinear model)

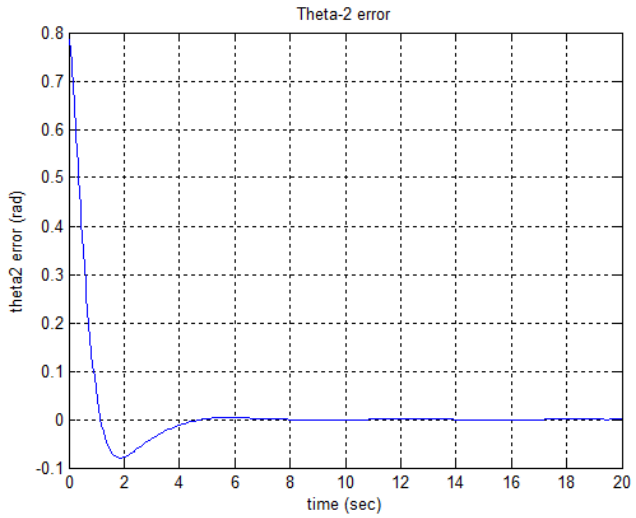

Fig. 14 : Error of theta_2 (Nonlinear model)

We notice that the response is following the control signal with relatively good manner. And errors of θ_1 and θ_2 are equal to zero in a short time.

VII. Conclusion

The main content of this paper is about modeling a 2-R robotusing two methods: first is mathematical modeling using Lagrange dynamic equations and the second is using Autodesk Inventor and Simulink software's to develop the model. After that we used PID controller to validate the models and to notice the difference in accuracy achieved by each technique. Linearization about working point is valid in one point only, while it is no longer valid for other points. The model designed from Autodesk Inventor and Simulink software's is giving better and reasonable response. Good results are found when using feedback linearization.

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Manuscript submission is a systematic procedure and little preparation is required beyond having all parts of your manuscript in a given format and a computer with an Internet connection and a Web browser. Full help and instructions are provided on-screen. As an author, you will be prompted for login and manuscript details as Field of Paper and then to upload your manuscript file(s) according to the instructions.

To avoid postal delays, all transaction is preferred by e-mail. A finished manuscript submission is confirmed by e-mail immediately and your paper enters the editorial process with no postal delays. When a conclusion is made about the publication of your paper by our Editorial Board, revisions can be submitted online with the same procedure, with an occasion to view and respond to all comments.

Complete support for both authors and co-author is provided.

4. MANUSCRIPT'S CATEGORY

Based on potential and nature, the manuscript can be categorized under the following heads:

Original research paper: Such papers are reports of high-level significant original research work.

Review papers: These are concise, significant but helpful and decisive topics for young researchers.

Research articles: These are handled with small investigation and applications

Research letters: The letters are small and concise comments on previously published matters.

5.STRUCTURE AND FORMAT OF MANUSCRIPT

The recommended size of original research paper is less than seven thousand words, review papers fewer than seven thousands words also.Preparation of research paper or how to write research paper, are major hurdle, while writing manuscript. The research articles and research letters should be fewer than three thousand words, the structure original research paper; sometime review paper should be as follows:

Papers: These are reports of significant research (typically less than 7000 words equivalent, including tables, figures, references), and comprise:

(a)Title should be relevant and commensurate with the theme of the paper.

(b) A brief Summary, "Abstract" (less than 150 words) containing the major results and conclusions.

(c) Up to ten keywords, that precisely identifies the paper's subject, purpose, and focus.

(d) An Introduction, giving necessary background excluding subheadings; objectives must be clearly declared.

(e) Resources and techniques with sufficient complete experimental details (wherever possible by reference) to permit repetition; sources of information must be given and numerical methods must be specified by reference, unless non-standard.

(f) Results should be presented concisely, by well-designed tables and/or figures; the same data may not be used in both; suitable statistical data should be given. All data must be obtained with attention to numerical detail in the planning stage. As reproduced design has been recognized to be important to experiments for a considerable time, the Editor has decided that any paper that appears not to have adequate numerical treatments of the data will be returned un-refereed;

(g) Discussion should cover the implications and consequences, not just recapitulating the results; conclusions should be summarizing.

(h) Brief Acknowledgements.

(i) References in the proper form.

Authors should very cautiously consider the preparation of papers to ensure that they communicate efficiently. Papers are much more likely to be accepted, if they are cautiously designed and laid out, contain few or no errors, are summarizing, and be conventional to the approach and instructions. They will in addition, be published with much less delays than those that require much technical and editorial correction.

The Editorial Board reserves the right to make literary corrections and to make suggestions to improve briefness.

It is vital, that authors take care in submitting a manuscript that is written in simple language and adheres to published guidelines.

Format

Language: The language of publication is UK English. Authors, for whom English is a second language, must have their manuscript efficiently edited by an English-speaking person before submission to make sure that, the English is of high excellence. It is preferable, that manuscripts should be professionally edited.

Standard Usage, Abbreviations, and Units: Spelling and hyphenation should be conventional to The Concise Oxford English Dictionary. Statistics and measurements should at all times be given in figures, e.g. 16 min, except for when the number begins a sentence. When the number does not refer to a unit of measurement it should be spelt in full unless, it is 160 or greater.

Abbreviations supposed to be used carefully. The abbreviated name or expression is supposed to be cited in full at first usage, followed by the conventional abbreviation in parentheses.

Metric SI units are supposed to generally be used excluding where they conflict with current practice or are confusing. For illustration, 1.4 l rather than 1.4 × 10-3 m3, or 4 mm somewhat than 4 × 10-3 m. Chemical formula and solutions must identify the form used, e.g. anhydrous or hydrated, and the concentration must be in clearly defined units. Common species names should be followed by underlines at the first mention. For following use the generic name should be constricted to a single letter, if it is clear.

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All manuscripts submitted to Global Journals Inc. (US), ought to include:

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Abstract, used in Original Papers and Reviews:

Optimizing Abstract for Search Engines

Many researchers searching for information online will use search engines such as Google, Yahoo or similar. By optimizing your paper for search engines, you will amplify the chance of someone finding it. This in turn will make it more likely to be viewed and/or cited in a further work. Global Journals Inc. (US) have compiled these guidelines to facilitate you to maximize the web-friendliness of the most public part of your paper.

Key Words

A major linchpin in research work for the writing research paper is the keyword search, which one will employ to find both library and Internet resources.

One must be persistent and creative in using keywords. An effective keyword search requires a strategy and planning a list of possible keywords and phrases to try.

Search engines for most searches, use Boolean searching, which is somewhat different from Internet searches. The Boolean search uses "operators," words (and, or, not, and near) that enable you to expand or narrow your affords. Tips for research paper while preparing research paper are very helpful guideline of research paper.

Choice of key words is first tool of tips to write research paper. Research paper writing is an art.A few tips for deciding as strategically as possible about keyword search:

- One should start brainstorming lists of possible keywords before even begin searching. Think about the most important concepts related to research work. Ask, "What words would a source have to include to be truly valuable in research paper?" Then consider synonyms for the important words.
- It may take the discovery of only one relevant paper to let steer in the right keyword direction because in most databases, the keywords under which a research paper is abstracted are listed with the paper.
- One should avoid outdated words.

Keywords are the key that opens a door to research work sources. Keyword searching is an art in which researcher's skills are bound to improve with experience and time.

Numerical Methods: Numerical methods used should be clear and, where appropriate, supported by references.

Acknowledgements: Please make these as concise as possible.

References

References follow the Harvard scheme of referencing. References in the text should cite the authors' names followed by the time of their publication, unless there are three or more authors when simply the first author's name is quoted followed by et al. unpublished work has to only be cited where necessary, and only in the text. Copies of references in press in other journals have to be supplied with submitted typescripts. It is necessary that all citations and references be carefully checked before submission, as mistakes or omissions will cause delays.

References to information on the World Wide Web can be given, but only if the information is available without charge to readers on an official site. Wikipedia and Similar websites are not allowed where anyone can change the information. Authors will be asked to make available electronic copies of the cited information for inclusion on the Global Journals Inc. (US) homepage at the judgment of the Editorial Board.

The Editorial Board and Global Journals Inc. (US) recommend that, citation of online-published papers and other material should be done via a DOI (digital object identifier). If an author cites anything, which does not have a DOI, they run the risk of the cited material not being noticeable.

The Editorial Board and Global Journals Inc. (US) recommend the use of a tool such as Reference Manager for reference management and formatting.

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Tables: Tables should be few in number, cautiously designed, uncrowned, and include only essential data. Each must have an Arabic number, e.g. Table 4, a self-explanatory caption and be on a separate sheet. Vertical lines should not be used.

Figures: Figures are supposed to be submitted as separate files. Always take in a citation in the text for each figure using Arabic numbers, e.g. Fig. 4. Artwork must be submitted online in electronic form by e-mailing them.

Preparation of Electronic Figures for Publication

Even though low quality images are sufficient for review purposes, print publication requires high quality images to prevent the final product being blurred or fuzzy. Submit (or e-mail) EPS (line art) or TIFF (halftone/photographs) files only. MS PowerPoint and Word Graphics are unsuitable for printed pictures. Do not use pixel-oriented software. Scans (TIFF only) should have a resolution of at least 350 dpi (halftone) or 700 to 1100 dpi (line drawings) in relation to the imitation size. Please give the data for figures in black and white or submit a Color Work Agreement Form. EPS files must be saved with fonts embedded (and with a TIFF preview, if possible).

For scanned images, the scanning resolution (at final image size) ought to be as follows to ensure good reproduction: line art: >650 dpi; halftones (including gel photographs) : >350 dpi; figures containing both halftone and line images: >650 dpi.

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6. AFTER ACCEPTANCE

Upon approval of a paper for publication, the manuscript will be forwarded to the dean, who is responsible for the publication of the Global Journals Inc. (US).

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(Free of charge) from the following website:

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TECHNIQUES FOR WRITING A GOOD QUALITY RESEARCH PAPER:

1. Choosing the topic: In most cases, the topic is searched by the interest of author but it can be also suggested by the guides. You can have several topics and then you can judge that in which topic or subject you are finding yourself most comfortable. This can be done by asking several questions to yourself, like Will I be able to carry our search in this area? Will I find all necessary recourses to accomplish the search? Will I be able to find all information in this field area? If the answer of these types of questions will be "Yes" then you can choose that topic. In most of the cases, you may have to conduct the surveys and have to visit several places because this field is related to Computer Science and Information Technology. Also, you may have to do a lot of work to find all rise and falls regarding the various data of that subject. Sometimes, detailed information plays a vital role, instead of short information.

2. Evaluators are human: First thing to remember that evaluators are also human being. They are not only meant for rejecting a paper. They are here to evaluate your paper. So, present your Best.

3. Think Like Evaluators: If you are in a confusion or getting demotivated that your paper will be accepted by evaluators or not, then think and try to evaluate your paper like an Evaluator. Try to understand that what an evaluator wants in your research paper and automatically you will have your answer.

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7. Use right software: Always use good quality software packages. If you are not capable to judge good software then you can lose quality of your paper unknowingly. There are various software programs available to help you, which you can get through Internet.

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10. Bookmarks are useful: When you read any book or magazine, you generally use bookmarks, right! It is a good habit, which helps to not to lose your continuity. You should always use bookmarks while searching on Internet also, which will make your search easier.

11. Revise what you wrote: When you write anything, always read it, summarize it and then finalize it.

12. Make all efforts: Make all efforts to mention what you are going to write in your paper. That means always have a good start. Try to mention everything in introduction, that what is the need of a particular research paper. Polish your work by good skill of writing and always give an evaluator, what he wants.

13. Have backups: When you are going to do any important thing like making research paper, you should always have backup copies of it either in your computer or in paper. This will help you to not to lose any of your important.

14. Produce good diagrams of your own: Always try to include good charts or diagrams in your paper to improve quality. Using several and unnecessary diagrams will degrade the quality of your paper by creating "hotchpotch." So always, try to make and include those diagrams, which are made by your own to improve readability and understandability of your paper.

15. Use of direct quotes: When you do research relevant to literature, history or current affairs then use of quotes become essential but if study is relevant to science then use of quotes is not preferable.

16. Use proper verb tense: Use proper verb tenses in your paper. Use past tense, to present those events that happened. Use present tense to indicate events that are going on. Use future tense to indicate future happening events. Use of improper and wrong tenses will confuse the evaluator. Avoid the sentences that are incomplete.

17. Never use online paper: If you are getting any paper on Internet, then never use it as your research paper because it might be possible that evaluator has already seen it or maybe it is outdated version.

18. Pick a good study spot: To do your research studies always try to pick a spot, which is quiet. Every spot is not for studies. Spot that suits you choose it and proceed further.

19. Know what you know: Always try to know, what you know by making objectives. Else, you will be confused and cannot achieve your target.

20. Use good quality grammar: Always use a good quality grammar and use words that will throw positive impact on evaluator. Use of good quality grammar does not mean to use tough words, that for each word the evaluator has to go through dictionary. Do not start sentence with a conjunction. Do not fragment sentences. Eliminate one-word sentences. Ignore passive voice. Do not ever use a big word when a diminutive one would suffice. Verbs have to be in agreement with their subjects. Prepositions are not expressions to finish sentences with. It is incorrect to ever divide an infinitive. Avoid clichés like the disease. Also, always shun irritating alliteration. Use language that is simple and straight forward. put together a neat summary.

21. Arrangement of information: Each section of the main body should start with an opening sentence and there should be a changeover at the end of the section. Give only valid and powerful arguments to your topic. You may also maintain your arguments with records.

22. Never start in last minute: Always start at right time and give enough time to research work. Leaving everything to the last minute will degrade your paper and spoil your work.

23. Multitasking in research is not good: Doing several things at the same time proves bad habit in case of research activity. Research is an area, where everything has a particular time slot. Divide your research work in parts and do particular part in particular time slot.

24. Never copy others' work: Never copy others' work and give it your name because if evaluator has seen it anywhere you will be in trouble.

25. Take proper rest and food: No matter how many hours you spend for your research activity, if you are not taking care of your health then all your efforts will be in vain. For a quality research, study is must, and this can be done by taking proper rest and food.

26. Go for seminars: Attend seminars if the topic is relevant to your research area. Utilize all your resources.

27. Refresh your mind after intervals: Try to give rest to your mind by listening to soft music or by sleeping in intervals. This will also improve your memory.

28. Make colleagues: Always try to make colleagues. No matter how sharper or intelligent you are, if you make colleagues you can have several ideas, which will be helpful for your research.

29. Think technically: Always think technically. If anything happens, then search its reasons, its benefits, and demerits.

30. Think and then print: When you will go to print your paper, notice that tables are not be split, headings are not detached from their descriptions, and page sequence is maintained.

31. Adding unnecessary information: Do not add unnecessary information, like, I have used MS Excel to draw graph. Do not add irrelevant and inappropriate material. These all will create superfluous. Foreign terminology and phrases are not apropos. One should NEVER take a broad view. Analogy in script is like feathers on a snake. Not at all use a large word when a very small one would be sufficient. Use words properly, regardless of how others use them. Remove quotations. Puns are for kids, not grunt readers. Amplification is a billion times of inferior quality than sarcasm.

32. Never oversimplify everything: To add material in your research paper, never go for oversimplification. This will definitely irritate the evaluator. Be more or less specific. Also too, by no means, ever use rhythmic redundancies. Contractions aren't essential and shouldn't be there used. Comparisons are as terrible as clichés. Give up ampersands and abbreviations, and so on. Remove commas, that are, not necessary. Parenthetical words however should be together with this in commas. Understatement is all the time the complete best way to put onward earth-shaking thoughts. Give a detailed literary review.

33. Report concluded results: Use concluded results. From raw data, filter the results and then conclude your studies based on measurements and observations taken. Significant figures and appropriate number of decimal places should be used. Parenthetical remarks are prohibitive. Proofread carefully at final stage. In the end give outline to your arguments. Spot out perspectives of further study of this subject. Justify your conclusion by at the bottom of them with sufficient justifications and examples.

34. After conclusion: Once you have concluded your research, the next most important step is to present your findings. Presentation is extremely important as it is the definite medium though which your research is going to be in print to the rest of the crowd. Care should be taken to categorize your thoughts well and present them in a logical and neat manner. A good quality research paper format is essential because it serves to highlight your research paper and bring to light all necessary aspects in your research.

Informal Guidelines of Research Paper Writing

Key points to remember:

- Submit all work in its final form.
- Write your paper in the form, which is presented in the guidelines using the template.
- Please note the criterion for grading the final paper by peer-reviewers.

Final Points:

A purpose of organizing a research paper is to let people to interpret your effort selectively. The journal requires the following sections, submitted in the order listed, each section to start on a new page.

The introduction will be compiled from reference matter and will reflect the design processes or outline of basis that direct you to make study. As you will carry out the process of study, the method and process section will be constructed as like that. The result segment will show related statistics in nearly sequential order and will direct the reviewers next to the similar intellectual paths throughout the data that you took to carry out your study. The discussion section will provide understanding of the data and projections as to the implication of the results. The use of good quality references all through the paper will give the effort trustworthiness by representing an alertness of prior workings.

Writing a research paper is not an easy job no matter how trouble-free the actual research or concept. Practice, excellent preparation, and controlled record keeping are the only means to make straightforward the progression.

General style:

Specific editorial column necessities for compliance of a manuscript will always take over from directions in these general guidelines.

To make a paper clear

· Adhere to recommended page limits

Mistakes to evade

- Insertion a title at the foot of a page with the subsequent text on the next page
- Separating a table/chart or figure - impound each figure/table to a single page
- Submitting a manuscript with pages out of sequence

In every sections of your document

· Use standard writing style including articles ("a", "the," etc.)

· Keep on paying attention on the research topic of the paper

· Use paragraphs to split each significant point (excluding for the abstract)

· Align the primary line of each section

· Present your points in sound order

· Use present tense to report well accepted

· Use past tense to describe specific results

· Shun familiar wording, don't address the reviewer directly, and don't use slang, slang language, or superlatives

· Shun use of extra pictures - include only those figures essential to presenting results

Title Page:

Choose a revealing title. It should be short. It should not have non-standard acronyms or abbreviations. It should not exceed two printed lines. It should include the name(s) and address (es) of all authors.

Abstract:

The summary should be two hundred words or less. It should briefly and clearly explain the key findings reported in the manuscript--must have precise statistics. It should not have abnormal acronyms or abbreviations. It should be logical in itself. Shun citing references at this point.

An abstract is a brief distinct paragraph summary of finished work or work in development. In a minute or less a reviewer can be taught the foundation behind the study, common approach to the problem, relevant results, and significant conclusions or new questions.

Write your summary when your paper is completed because how can you write the summary of anything which is not yet written? Wealth of terminology is very essential in abstract. Yet, use comprehensive sentences and do not let go readability for briefness. You can maintain it succinct by phrasing sentences so that they provide more than lone rationale. The author can at this moment go straight to shortening the outcome. Sum up the study, with the subsequent elements in any summary. Try to maintain the initial two items to no more than one ruling each.

- Reason of the study - theory, overall issue, purpose
- Fundamental goal
- To the point depiction of the research
- Consequences, including definite statistics - if the consequences are quantitative in nature, account quantitative data; results of any numerical analysis should be reported
- Significant conclusions or questions that track from the research(es)

Approach:

- Single section, and succinct
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- A conceptual should situate on its own, and not submit to any other part of the paper such as a form or table
- Center on shortening results - bound background information to a verdict or two, if completely necessary
- What you account in an conceptual must be regular with what you reported in the manuscript
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Introduction:

The **Introduction** should "introduce" the manuscript. The reviewer should be presented with sufficient background information to be capable to comprehend and calculate the purpose of your study without having to submit to other works. The basis for the study should be offered. Give most important references but shun difficult to make a comprehensive appraisal of the topic. In the introduction, describe the problem visibly. If the problem is not acknowledged in a logical, reasonable way, the reviewer will have no attention in your result. Speak in common terms about techniques used to explain the problem, if needed, but do not present any particulars about the protocols here. Following approach can create a valuable beginning:

- Explain the value (significance) of the study
- Shield the model - why did you employ this particular system or method? What is its compensation? You strength remark on its appropriateness from a abstract point of vision as well as point out sensible reasons for using it.
- Present a justification. Status your particular theory (es) or aim(s), and describe the logic that led you to choose them.
- Very for a short time explain the tentative propose and how it skilled the declared objectives.

Approach:

- Use past tense except for when referring to recognized facts. After all, the manuscript will be submitted after the entire job is done.
- Sort out your thoughts; manufacture one key point with every section. If you make the four points listed above, you will need a least of four paragraphs.

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This part is supposed to be the easiest to carve if you have good skills. A sound written Procedures segment allows a capable scientist to replacement your results. Present precise information about your supplies. The suppliers and clarity of reagents can be helpful bits of information. Present methods in sequential order but linked methodologies can be grouped as a segment. Be concise when relating the protocols. Attempt for the least amount of information that would permit another capable scientist to spare your outcome but be cautious that vital information is integrated. The use of subheadings is suggested and ought to be synchronized with the results section. When a technique is used that has been well described in another object, mention the specific item describing a way but draw the basic principle while stating the situation. The purpose is to text all particular resources and broad procedures, so that another person may use some or all of the methods in one more study or referee the scientific value of your work. It is not to be a step by step report of the whole thing you did, nor is a methods section a set of orders.

Materials:

- Explain materials individually only if the study is so complex that it saves liberty this way.
- Embrace particular materials, and any tools or provisions that are not frequently found in laboratories.
- Do not take in frequently found.
- If use of a definite type of tools.
- Materials may be reported in a part section or else they may be recognized along with your measures.

Methods:

- Report the method (not particulars of each process that engaged the same methodology)
- Describe the method entirely
- To be succinct, present methods under headings dedicated to specific dealings or groups of measures
- Simplify - details how procedures were completed not how they were exclusively performed on a particular day.
- If well known procedures were used, account the procedure by name, possibly with reference, and that's all.

Approach:

- It is embarrassed or not possible to use vigorous voice when documenting methods with no using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result when script up the methods most authors use third person passive voice.
- Use standard style in this and in every other part of the paper - avoid familiar lists, and use full sentences.

What to keep away from

- Resources and methods are not a set of information.
- Skip all descriptive information and surroundings - save it for the argument.
- Leave out information that is immaterial to a third party.

Results:

The principle of a results segment is to present and demonstrate your conclusion. Create this part a entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently.You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.

Content

- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or in manuscript form.

What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
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- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
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- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

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References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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