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## Preface

We would like to present, with great pleasure, the inaugural volume-6, Issue-10, October 2020, of a scholarly journal, *International Journal of Engineering Research & Science*. This journal is part of the AD Publications series *in the field of Engineering, Mathematics, Physics, Chemistry and science Research Development*, and is devoted to the gamut of Engineering and Science issues, from theoretical aspects to application-dependent studies and the validation of emerging technologies.

This journal was envisioned and founded to represent the growing needs of Engineering and Science as an emerging and increasingly vital field, now widely recognized as an integral part of scientific and technical investigations. Its mission is to become a voice of the Engineering and Science community, addressing researchers and practitioners in below areas

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Each article in this issue provides an example of a concrete industrial application or a case study of the presented methodology to amplify the impact of the contribution. We are very thankful to everybody within that community who supported the idea of creating a new Research with IJOER. We are certain that this issue will be followed by many others, reporting new developments in the Engineering and Science field. This issue would not have been possible without the great support of the Reviewer, Editorial Board members and also with our Advisory Board Members, and we would like to express our sincere thanks to all of them. We would also like to express our gratitude to the editorial staff of AD Publications, who supported us at every stage of the project. It is our hope that this fine collection of articles will be a valuable resource for *IJOER* readers and will stimulate further research into the vibrant area of Engineering and Science Research.

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## Synthesis Methods of Saccharin-6-Carboxylic Acid Triglyceride E.T. Aslanova

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**Abstract**— One and two-step methods for the synthesis of saccharin-6-carboxylic acid triglyceride were studied. The reesterification reactions of 2-hydroxypropyl-1,3-bis-ethersulfoimide of this acid and glycerol with some aliphatic saccharin-6-carboxylic acid esters were carried out. The resulting products are characterized by elemental analysis and IR spectroscopy and size exclusion chromatography. It was found that when using a two-step method, the end product is obtained with the highest yield (85%).

Keywords— alkyl esters, glycerine, IR-spectra, monomer, reesterification, saccharin-6-carboxylic acid.

#### I. INTRODUCTION

Aromatic and aliphatic polysulfoimides (polysaccharines) are sulfur-containing analogues of polyimides. Polysulfoimides differ from polyimides by the presence in the imide cycle of one carbonyl (C=O) and one sulfonyl ( $-SO_2$ -) group [1]. These polymers possess high thermal stability (500-650°C), solubility, stability to radiation and light, acid and alkaline hydrolysis [2].

Polymer composite materials made based on compounds which contain epoxy and sulfoimide groups have high physicomechanical and thermal properties [3]. Epoxyimide oligomers possess the highest heat resistance, in which glycidyl groups are directly adjacent to the imide cycle [4]. In this regard, they are widely used as high-strength and heat-resistant construction materials.

It is known that epoxy resins, in particular dianes, are used after the introduction of hardening agents. Considering that the majority of epoxy composite materials in hardened form have brittleness that impairs the physicomechanical properties of the obtained material, often compounds with a plasticizing property are introduced into the composition, for example, esters and compounds containing groups capable of hardening by epoxy groups of the resin [5-7].

In this regard, given the advantages of polysaccharines, there is a need to develop effective methods for the synthesis of new monomers (prepolymers) based on saccharin monocarboxylic acids [8]. There is evidence of the use of diimidodicarboxylic acids as monomers in the synthesis of polyetherimides. The properties of these polymers can change due to the introduction of various fragment groups into their molecule [9, 10].

There is data in the literature on the synthesis of diesterodisulfoimides by reesterification of the methyl ester of saccharinmonocarboxylic acids with aliphatic and aromatic glycols [11, 12].

This paper describes one- and two-step methods for the synthesis of saccharin-6-carboxylic acid triglyceride.

The aim of the work is the synthesis of new monomers based on saccharin-6-carboxylic acid, for their further use in the preparation of highly branched and net heat-resistant polymers.

#### II. EXPERIMENTAL

#### 2.1 Synthesis of triglyceride based on 2-hydroxypropyl-1,3-bis-ethersulfoimide saccharin-6-carboxylic acid

A mixture of 20 g (0.039 mol) of 2-hydroxypropyl-1,3-bis-etirosulfoimide of saccharin-6-carboxylic acid, 9.53 g (0.039 mol) of saccharin-6-carboxylic acid methyl ester and 2 g of PbO in 200 ml of DMF at stirring was heated to 124°C for 2 hours. The reaction mass was washed with acetone, the end product was isolated from the reaction medium as a precipitate by precipitation with distilled water.

The obtained coffee-milk colour powder product was dried at room temperature and brought to constant weight in vacuum.  $T_{\rm m} = 167^{\circ}$ C.

Similarly, the above mentioned compounds were synthesized based on saccharin-6-carboxylic acid ethyl and isopropyl esters.

#### 2.2 Synthesis of triglyceride by reesterification of aliphatic esters of saccharin-6-carboxylic acid with glycerine

A mixture of 40 g (0.016 mol) of saccharin-6-carboxylic acid methyl ester, 7.5 g of PbO in 4.6 g (0.05 mol) of propane-1,2,3-triol (glycerine) was heated to 115-117°C with stirring for 2 hours. The reaction mass was washed with acetone, and then was isolated as a precipitate by precipitation with distilled water. The obtained coffee-milk colour powder product was dried at room temperature and brought to constant weight in vacuum. Tm =167°C.

In the work, distilled glycerine of the PK-94 brand was used with a mass fraction of pure glycerine of not less than 94.0%. Physicochemical properties of glycerine and solvents are corresponded to the literature data [13].

Infrared spectra were recorded on an infrared Fourier spectrometer LUMOS (firm BRUKER Germany) in the wavelength range of 600-4000 cm<sup>-1</sup>, using an ATR attachment with a ZnSe crystal. The diameter of the crystal is 1 cm. The number of scans of the sample is 24; the measurement duration is 30 seconds.

Elemental analysis was performed according to the method [14], based on the pyrolytic burning of organic matter in an oxygen stream using the Pregl apparatus.

Molecular weights (MW) and molecular weight distribution (MWD) parameters of the synthesized products were determined by size exclusion chromatography (SEC) on the high performance liquid chromatograph (*Kovo, Czech Republic*) with a refractometric detector. Two columns with a size of  $3.3 \times 150$  mm filled with a stationary Separon-SGX phase with a particle size of 7 µm and a porosity of 100 Å were used. Eluent – DMF, flow rate 0.3 ml/min. T=20-25°C. The calibration dependence of *lgM* on the hold-up volume *V<sub>R</sub>* in the range M=(1.5-100)×10<sup>2</sup>, described by the equation V<sub>R</sub>=C<sub>1</sub>-C<sub>2</sub>lgM, where C<sub>1</sub>=24.4, C<sub>2</sub>=4, was obtained using PRG standards. The interpretation of MMP chromatograms was performed according to the method [15, 16]. The calculations are carried out according to the equations:

$$M_{w} = \sum M_{i} \omega_{i}; M_{n} = 1/\omega_{i} / \sum M_{i}$$

$$\tag{1}$$

where  $M_w$  – weight-average molecular weight,  $M_n$  – number-average molecular weight,  $M_i$  –molecular weight corresponding to the *i*<sup>th</sup> area of the chromatogram,  $\omega_i$  – fraction of the area of part *i*.

#### III. RESULTS AND DISCUSSION

For the synthesis of triglyceride of saccharin-6-carboxylic acid was first applied a method of obtaining on the basis of our previously synthesized 2-hydroxypropyl-1,3-bis-ethersulfoimide saccharin-6-carboxylic acid [17].

The process was carried out in two steps.

In the first step, 2-hydroxypropyl-1,3-bis-ethersulfoimide of saccharin-6-carboxylic acid was obtained:



 $R = -CH_3, -C_2H_5, -C_3H_7$ 

As a result of reesterification, 2-hydroxypropyl-1,3-bis-ether-sulfosulfoimide of saccharin-6-carboxylic acid was obtained, which included ester and sulfoimide groups [18]. The yield of the end product is 85%.

It was found that the IR spectra of this compound (Fig. 1) contain absorption bands in the regions of 1385, 1434, 1454 cm<sup>-1</sup> deformation and 2851, 2884, 2921 cm<sup>-1</sup> valence vibrations of C–H bonds of the CH<sub>3</sub> and <sub>CH2</sub> groups; deformation vibrations (673, 713, 755, 829 cm<sup>-1</sup>) of C–H bond of the substituted benzene ring; deformational vibrations (1487, 1605 cm<sup>-1</sup>) of C–H

bond of the benzene ring; valence vibrations (1039cm<sup>-1</sup>) of C–O bonds of the alcohol; valence vibrations (1719 cm<sup>-1</sup>) of the C=O group of the ester; valence vibrations (1166, 1244 cm<sup>-1</sup>) C–O bond of the ester bond; valence vibrations (1129, 1146, 1278, 1290 cm<sup>-1</sup>) of SO<sub>2</sub> groups; and absorption bands at 1643, 1553, 1339 cm<sup>-1</sup> – amide bands for the H–N–C=O group [19, 20].



FIGURE 1: IR spectra of 2-hydroxypropyl-1,3-bis-ether-sulfosulfoimide of saccharin-6-carboxylic acid

In the second step, by the interaction of the obtained 2-hydroxypropyl-1,3-bis ethersulfoimide with esters of the same acid under more severe conditions, saccharin-6-carboxylic acid triglyceride was synthesized. The reaction proceeded as follows:



$$R = -CH_3, -C_2H_5, -C_3H_7$$

The yield of the end product is 85%.

It was found that the obtained product is a light coffee-milk colour powder, soluble only in aprotic solvents such as DMF, DMAA, DMSO, etc.

The composition and structure of the obtained compound was determined by elemental analysis (Table 1) and IR spectroscopy.

TABLE 1
PHYSICAL CONSTANTS OF SACCHARIN-6-CARBOXYLIC ACID TRIGLYCERIDE SYNTHESIZED BY A TWO-STEP
METHOD

Compound	Empirical formula	Found,% Calculated,%				М	T <sub>m</sub> ,°C	Yield, %
		С	Η	Ν	S			
saccharin-6-carboxylic acid triglyceride	CarH17O15N2S2	44.95	2.48	5.81	13.43	719	167	85
	02/11/0151303	45.06	2.36	5.84	13.35	,1)	107	05

In the IR spectra of the obtained compound (Fig. 2), deformation absorption bands appear in the 1385, 1438, 1485 cm<sup>-1</sup> region of the C–H bond of the CH<sub>2</sub> groups; valence vibrations (1643 cm<sup>-1</sup>) of the C=O bond of the amide; valence vibrations (1719 cm<sup>-1</sup>) C=O of the ester bond; valence vibrations (1108, 1129, 1147, 1168 cm<sup>-1</sup>) of the C–O bond of the ester; valence vibrations (1252, 1277 cm<sup>-1</sup>) of the SO<sub>2</sub> group; deformation (1553 cm<sup>-1</sup>) and valence (3268, 3352 cm<sup>-1</sup>) vibrations of the N–H bond; deformation (674, 694, 713, 757, 788, 829, 867, 1607 cm<sup>-1</sup>) vibrations of the C–H bond of the substituted benzene ring.



#### FIGURE 2: IR spectra of saccharin-6-carboxylic acid triglyceride synthesized by a two-step method

The one-step synthesis method was carried out by reesterification of saccharin-6-carboxylic acid alkyl esters with propane-1,2,3-triol (glycerol) according to the following scheme:



 $R = -CH_3, -C_2H_5, -C_3H_7$ 

The obtained product is a light coffee-milk colour powder, soluble only in aprotic solvents such as DMF, DMAA, DMSO, etc.

The composition and structure of the obtained compound was determined by elemental analysis (Table 2) and IR spectroscopy.

 TABLE 2

 PHYSICAL CONSTANTS of SACCHARIN-6-CARBOXYLIC ACID TRIGLYCERIDE SYNTHESIZED BY a ONE-STEP

 METHOD

Compound	Empirical formula	Found, % Calculated, %				М	T <sub>m</sub> ,°C	Yield, %
	101 muta	С	Н	Ν	S			
saccharin-6-carboxylic acid triglyceride	$C_{27}H_{17}O_{15}N_3S_3$	$\frac{44.98}{45.06}$	$\frac{2.46}{2.36}$	$\frac{5.79}{5.84}$	$\frac{13.31}{13.35}$	719	167	67

The following absorption bands were observed in the IR spectra (Fig. 3) of the obtained compound: deformation (720, 395, 1454 cm<sup>-1</sup>) and valence vibrations of the C–H bond of CH<sub>2</sub> groups; valence vibrations (1644 cm<sup>-1</sup>) of the C=O bond of the amide; valence vibrations (1719 cm<sup>-1</sup>) of the C=O bond of the ester; valence vibrations (1153 cm<sup>-1</sup>) of the C–O bond of the ester; valence vibrations (1239, 1283 cm<sup>-1</sup>) of the SO<sub>2</sub> group; valence vibrations (1019 cm<sup>-1</sup>) of the S=O bond; deformation (1573 cm<sup>-1</sup>) and valence vibrations (3276 cm<sup>-1</sup>) of the N–H bond; deformation vibrations (617, 678, 750, 859, 1607 cm<sup>-1</sup>) of the C–H bond of the substituted benzene ring.



FIGURE 3: IR spectra of saccharin-6-carboxylic acid triglyceride synthesized by a one-step method

The identity of the end product is confirmed through molecular weight (MW) by size-exclusion chromatography (Fig. 4). Identical peaks were obtained for triglycerides synthesized by various methods. So the peak recorded at  $V_R = 15$  according to MW corresponds to 220 ( $M_{\text{theoretical}}$  222) ( $\lg M=2.35$ ).



#### FIGURE 4: MWD exclusion curve of saccharin-6-carboxylic acid triglyceride. Chromatography conditions: Columns 3.3 × 150 mm, adsorbent: Separon – SGX with a particle size of 7 μm and a porosity of 100 Å. Eluent – DMF, flow rate 0.3 ml / min. Detector: refractometer. 1 count = 0.13 ml

It should be noted that in order to select the optimal temperature-time regime, the above reaction was carried out under various conditions. With increasing temperature or reaction time, oligomeric products of the same composition were obtained.

#### IV. CONCLUSION

Thus, based on the research, the following conclusions can be drawn. One- and two-step methods for the synthesis of saccharin-6-carboxylic acid triglyceride have been developed; it was found that in the synthesis of triglyceride by the two-step method, the end product is obtained with the highest yield -85%; the synthesized saccharin-6-carboxylic acid triglyceride is of interest for its use as a monomer in the production of highly branched and cross-linked sulfoimide-containing polymers and epoxy resins, and also as a hardener-plasticizer of industrial epoxy resins.

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# The Test of Permeable by Expansion Valve in Cooling **Compressor** Ľubica Bednárová<sup>1</sup>, Lukaš Tóth<sup>2</sup>, Filip Duda<sup>3</sup>, Ľubomíra Kmeťová<sup>4</sup>

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**Abstract**— Current article describes the HFC test, which the measure the amount of impurity in cooling compressors, which they could cause damage to them. In introduce of the article describes the process of cooling cycle in cooling equipment. In the tab. 1 are summarized the basic advantages and disadvantages of use natural based refrigerant, which are used in producing the compressors designated for equipment's as refrigerators, air conditions, cooling rooms. In the article are describe the media, which it may occur in compressor, but their presence can may cause the problems with the operation of compressor possibly its damage. We say about media respectively the products, which they are not miscible with oil. It is water, paraffin, silicone and alkali products. Test HFC discovers and measurements the presence these components and on the basis of their quantity decides on the quality of production of refrigeration compressors. The article describes the test procedure and in the end the evaluation of the maximum value of the given incompatible respectively unwanted products.

#### Keywords— compressor, HFC test, refrigerant, refrigeration equipment.

#### I. INTRODUCTION

In the past the cooling was used in particular to cooling of food. At present, the demand for refrigeration technology is the much larger. Households use refrigeration not only for food storage but also in air conditioning units. In the chemical industry, refrigeration is used to extract rare gases. In the medical environment, cooling is an essential aspect of storing transfused blood. Mechanical engineering uses cooling in various work processes, such as cutting. Thanks to cooling, the performance of cutting tools up to 100%. This could be continued and mention many industries for which refrigeration is an integral part [1].

One of the main parts of the refrigeration equipment is the compressor, which ensures the circulation of the refrigerant. The cooling circuit consists of four basic processes (fig. 1):



FIGURE 1: Compressor cooling circuit

Refrigerant flows through the refrigeration system, which changes its state, temperature and pressure.

1) The heart of an air conditioner is a compressor that "pumps" the refrigerant in the circuit.

- 2) Before the compressor, the refrigerant vapors have a low pressure; the compressor compresses these vapors to a high pressure, while the vapors also heat up.
- 3) High temperature and pressure vapors flow to the condenser, where they transfer heat to the surrounding air and liquefy. Liquid refrigerant leaves the condenser.
- 4) Liquid refrigerant still at high pressure flows through the expansion valve, which reduces the pressure and thus the temperature of the liquid refrigerant to a lower temperature than the refrigerated space.
- 5) Cold liquid refrigerant with low pressure flows into the evaporator, where it removes heat from the environment by evaporation. The refrigerant evaporates completely and the low-pressure refrigerant vapours are sucked in by a compressor, which ensures its circulation (Fig. 2).



## FIGURE 2: Schematic representation of the cooling circuit [3]

#### II. COMPRESSORS OVERVIEW

Fixed and variable speed hermetic compressors are available on the market for domestic refrigerators, freezers and minirefrigerators, as well as refrigerated display cases and industrial refrigerated boxes, walk-in refrigerators, ice makers, refrigerated counters, tables, rooms and much more.

When choosing a refrigerant for a certain type of compressor, its thermodynamic properties, physical and chemical properties are decisive, including solubility with water and oils, impact on construction materials, impact on the human body, ecological properties and last but not least price and availability[2].

Currently, the company places great emphasis on ecological operation with the least possible impact on the environment. Most compressors are designed to use natural refrigerants, with the least possible environmental impacts such as ozone depletion, the greenhouse effect and various photochemical reactions. Therefore, the types of refrigerants belonging to the group of fluorocarbons are used for the production of compressors, namely refrigerant R 134a a R 600a. The following Tab. 1 summarizes the characteristics of the selected oils.

Oil type	Cooling	Advantages	Disadvantages	
Mineral oil - paraffinic	Ammonia	Medium thermal stability, viscosity index is higher.	High flocculation point, low viscosity and miscibility, wax separation.	
Raftenic mineral oil	R12, R22, R403, ammonia, R290, R600a	Good viscosity, lower viscosity index, low flocculation point, good miscibility, good heart rate. chem. stability, low foaming.	2x more expensive than mineral oils.	
Alkyl benzene oil	R12, R22, R403	Good thermo-chemical stability, low foaming, high viscosity index.	Poor miscibility. 5 times more expensive than mineral oils.	
Polyalkylene glycols (PAG)	Ammonia	Good lubricity, high viscosity index.	High hygroscopicity	
Polyol ester (POE)	HFC cooling: R134a, R507, R23, R404a	Low flocculation point, good thermochemical stability, low foaming, medium to high viscosity index.	Limited miscibility at low temperatures, higher hygroscopicity.	

 TABLE 1

 Types of oils and refrigerants used in the manufacture of refrigeration compressors

The biggest problem for introducing these oils and refrigerants into the production process is the effect of the adaptation of industrial residues to gas. Residues such as oil, which protects the components used in the manufacture of compressors against oxidation, moisturizer used by production workers and other incompatible products, endanger the quality of refrigerant products because they are not miscible with the oil and refrigerant. They react or form products that react with the refrigerant, oil, compressor components, and compressor residues and thus change its operating characteristics, negatively affecting the performance of the compressor. It reacts or forms products that react with the refrigerant, oil, compressor residues and thus change its operating characteristics, negatively affecting the performance of the compressor. It reacts or forms products that react with the refrigerant, oil, compressor components, and thus change its operating characteristics, negatively affecting the performance of the compressor.

#### III. PRODUCTS THAT ARE NOT MISCIBLE WITH OIL COOLING MEDIUM



#### 3.1 Water

- Induces the formation of acids by reaction with oil (organic carboxylic acid) or with refrigerant (hydrochloric acid), then reacts with the metal components of the compressor, minerals are formed, insulating materials in the compressor are degraded.
- ➢ Freezes like an ice drop.

#### **3.2** Alkaline products (degreasers)

- > They cause the formation of salts of carboxylic acids by reaction with oil.
- > They cause the reverse reaction of the polyester oil synthesis, thus creating an acidic environment in the compressor.

#### 3.3 Paraffin

At higher temperatures it is soluble in refrigerant and in polyester oil, at lower temperatures it is insoluble and clogs the expansion valve.

#### 3.3.1 Sources

- Preservative oils.
- Oils used in pressing,

- > Paraffin was used as a slip driver on the wire for winding electric motors.
- Protective hand creams.
- Grease machining emulsions.
- Maintenance products e.g.vaseline.

#### 3.4 Silicone oils

At higher temperatures they are soluble in refrigerant and polyester oil, at lower temperatures it is insoluble and clogs the capillary.

#### 3.4.1 Sources

- > They are part of molding separators.
- Parts are of adhesives, sealants.
- > They are used as modeling agents in the production of plastic components.
- Maintenance products.

#### IV. TEST HFC

HFC stands for H-hydrogen, F-fluorine and C-carbon. This special test is performed to determine the amount of contamination of the mechanics and the compressor space. Chemical reactions of several substances can also occur during compressor operation. The cleanliness of the compressor production line is very important for this test. During the operation of the compressor, chemical reactions of several substances occur, which can affect the life and quality of the compressor. It is the HFC test that determines the type and amount of residues (residues) in the expansion valve and in the evaporator.

During the test, three compressors are tested at the same time (Fig. 3). For new types of compressors that have not yet been tested, it is necessary to subject the compressor to measurements on a calorimeter, where the dependence of the stator winding temperature and the casing is determined. When the whole system is ready, the compressor is cleaned in a chemical laboratory. Here, the entire pipe, together with the evaporator and condenser, is flushed with petroleum ether. At the same time, the analysis of soluble and insoluble residues is processed. This procedure removes cutting residues or other contaminants that could contaminate the compressor system. The components, including the exchanger, are dried at  $100 \degree C$  for 3 hours. Follow, the diameter measurement of the expansion valve with nitrogen. Subsequently, the system is assembled and welded without borax additives. A leak test and vacuuming follow. If no leaks are detected in the system, the compressor starts running for 500 to 1000 hours. In Fig. 4 the compressor in the test device is connected.





FIGURE 3: Refrigeration compressor testing equipment

FIGURE 4: Compressor connection in the test rig

At the end of the test, the system is cut near the compressor and an oil sample is taken immediately. The bottle to be sampled must be completely filled to prevent contamination of the oil with air. Subsequently, the expansion valve is carefully removed so as not to damage its inner diameter. The capillary diameter is then measured using nitrogen. Other components, including the capillary, are again tested in a chemical laboratory. The expansion valve is cleaned again to measure the diameter after the test.

#### V. CONCLUSION

All results are processed in a capillary flow restriction report. It is developed a chemical control and work with wear analysis. In Fig. 5 shows a section through a capillary where the deposition of paraffin in the capillaries is affected and also the grey deposited dust, which may affect the life of the compressor.

The HFC test is designed to detect the effect of various components on cooling. Chemical reactions that occur during compression can cause it to fail. It is therefore necessary to ensure such conditions in the manufacture of the compressor that contamination of the internal parts of the compressor did not occur. It is very important to pay attention not only to the cleanliness of the plant, but also to work equipment and protective equipment, which should not have substances that can cause an undesirable chemical reaction with refrigeration or oil. The HFC test detects the amount of increasing impurities in the expansion valve and in the evaporator.



**FIGURE 5: Section though a capillary** 

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## **Comparing Oxygen Plasma, Hydrogen Peroxide and Flame Treatments of Polyamide Tubes to Hydrophilic Coating Adhesion**

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**Abstract**— Hydrophilic-coated catheters reduce the endovascular friction between the device and blood and increase the biocompatibility of catheters. This study investigated three surface treatments to improve catheter surface. Oxygen plasma, hydrogen peroxide, and flame exposure coating equipment were manufactured. Polyamide 11 (PA11) catheter surfaces were treated and the influence of exposure time for each individual technique was studied. Treated samples were characterized by goniometry, scanning electron microscopy, infrared spectroscopy and scratch resistance of the hydrophilic coating deposited. Oxygen plasma, hydrogen peroxide, and flame treatment during 60 s showed significant improvement in surface properties, in which both wettability and the adhesion of the hydrophilic coating were superior in comparison to the other samples. The devices developed in these work were able to coat and improve catheter surface properties.

Keywords— Oxygen Plasma, Hydrogen Peroxide, Flame Treatments, Polyamide Tubes, Hydrophilic Coating.

#### I. INTRODUCTION

The incorporation of minimally invasive practices in surgical procedures has provided many facilities and benefits compared to equivalent traditional surgical techniques1. Through small localized incisions and using specific equipment, surgical management is restricted only to the diseased area and thus preserves the peripheral structures. This reduces the need for blood replacement, the length of hospital internment, the likelihood of infections, and the postoperative pain  $1^{-6}$ . However, these new techniques also have some disadvantages. In the case of endovascular procedures one of the biggest problems is the trauma and injury caused to blood vessel walls due to friction during the introduction of the device<sup>2,3,5,6</sup>. Hydrophiliccoated catheters have contributed to further broadening the range of benefits offered by minimally invasive surgery through a significant reduction in the endovascular friction between the device and blood, as well as an increase in the biocompatibility of catheters. Hydrophilic coatings are polar materials and have high affinity for liquids of the same nature as blood, making the interface between them slide more easily<sup>3</sup>. Moreover, the hydrophilic coatings have given positive results in tests for cytotoxicity, systemic toxicity, and acute intracutaneous reactivity, among others, demonstrating that they also satisfy requirements related to biocompatibility<sup>2,3</sup>. Generally, methods to obtain a hydrophilic polymeric coating on another polymer component or device can be divided into three stages: (i) treatment of the device surface, (ii) deposition of the coating and (iii) curing of the coating. The surface treatment is necessary mainly due to a difference in polarity between the hydrophilic coating and the polymer device, factor which hinder the adhesion between them. The surface treatment of 7 polymeric devices can be accomplished by various methods ranging from wet to dry processes such as controlled oxidation using oxyacids<sup>6-8</sup>, plasma<sup>9-12</sup> and flame treatments<sup>13</sup>. Generally, techniques for surface modification are used to introduce polar functional groups that promote interaction at the interface between the device and polymer coating through intermolecular interactions, improving the wettability of the device<sup>3,8,12,14</sup>. This study investigated the use of oxygen plasma, hydrogen peroxide solution and flame exposure treatments to provide hydrophilicity to polyamide 11 (PA11) catheter surfaces. In particularly, the influence of exposure 64 times for each individual technique was evaluated with the aim of increasing the adhesion between the PA11 catheter and the hydrophilic coating. The wettability and adhesion of a hydrophilic coating were investigated. Treated samples were characterized by goniometry, scanning electron microscopy, infrared spectroscopy and scratch resistance of the hydrophilic coating deposited.

#### II. EXPERIMENTAL

#### 2.1 Materials

Medical grade catheters of pure PA11 (AP Extrusion Incorporated, USA) with 1,130 mm of length, 3.8 mm of outside diameter and 0.6 mm of wall thickness were used as substrates in the present study. The catheters were cut to test samples with 40 mm of length and then washed with ethanol. The hydrophilic polyurethane oligomer based on aromatic polyisocyanate (Hypol JM, Dow Chemical Company, USA) was used as hydrophilic coating on PA11 samples surfaces.

#### 2.2 Oxygen plasma treatment

An AC electrical discharge reactor, with a frequency of 50 Hz, was employed for the oxygen plasma treatment of the samples. Two aluminum electrodes were arranged in parallel inside the reactor. The electrical apparatus supplies a voltage of 2 kV at these electrodes, with 20 mA current maximum. Oxygen was confined in the reactor and submitted to pressures of around 8x10-1 Pa. The electric field created between the electrodes accelerates the gas particles, causing a chain reaction and increasing the concentration of ionized species. In this way the low temperature plasma (LTP) is generated. The exposure times were 1, 2, 3, 5 and 8 min.

#### 2.3 Hydrogen peroxide treatment

A reflux system was employed for the hydrogen 95 peroxide treatment of the samples. An aqueous solution containing 20% of hydrogen peroxide was placed in a glass flask and closed. An electrical resistance was used to heat this solution to boiling point (102°C). The samples were fully submerged in this solution and treated one at a time. The exposure times were 0.5, 1, 2, and 4 h. Before characterization all samples were dried with paper towels.

#### 2.4 Flame treatment

An apparatus built in the laboratory was used for surface treatment of the samples by intermittent exposure to the flame. The samples were held in the shaft and rotated perpendicularly to the flame of the torch to a constant frequency of 73 rpm. The exposure times were set at 5, 10, 20, 30 and 60 s to avoid causing changes, which could adversely affect the sample surface during treatment 13. The surface temperature of the samples was monitored by infrared thermometer. Because of the peculiarities of each technique, samples were previously evaluated by exposure to different times to define the optimal time interval for each technique investigation. The main criterion adopted was defining time range in which the surface of the samples showed no significant visual changes in sample color.

#### 2.5 Contact Angle Determination

The technique of goniometry was used for the analysis of the variation in the contact angle as function of treatment time for the samples, at least 4h after the surface treatments. The equipment used was a Data Physics goniometer, model OCA-15, operated at ambient temperature and pressure. The contact angle was measured by the sessile drop method based on ASTM D724 - 99 (2003) and ASTM D5725 - 99 (2008). The radius of the base (b) and height (h) of the drop were measured and the contact angle was calculated using equation 12:

Contact angle (
$$\theta$$
) = arcsin $\left[\frac{2bh}{b^2+h^2}\right]$  (1)

Two drops of deionized water were deposited for each time established in all the techniques, and two contact angles were measured per drop. The image of each drop was captured by the camera's high-resolution device connected to a computer.

#### 2.6 Scanning Electron Microscopy (SEM) Analysis

For the image analysis by scanning electron microscopy of the PA11 samples, as well as the analysis of the scratch resistance of the samples coated with hydrophilic polymer, a XL-30 Philips scanning electron microscope was used. Before the tests, all samples were coated with a thin layer (20 nm) of gold.

#### 2.7 Fourier Transform Infrared (FTIR) Analyses

The FTIR technique was used in reflectance mode (ATR) for the evaluation of possible chemical changes in the samples after treatment. The device used was a Perkin Elmer infrared spectrometer. The spectra generated were obtained from scans of the samples in the region from 3500 to 800 cm-1, at 4 cm-1 resolution.

#### 2.8 Application of Hydrophilic Coating on PA11 Samples and Scratch Resistance Test

For a uniform application of the hydrophilic polymer on the surface of the PA11 tube samples the device shown in the diagram in Figure 1 was developed. The samples were fixed to a metal rod, which in turn was connected to the rotor shaft. While the sample was spinning at a constant speed, the hydrophilic polymer was applied on the surface until they were fully covered. The blade 0.1 mm from the surface uniformly regulated the thickness of the layer deposited. After the coating, the tubes were left rotating in the device for an hour for curing of the hydrophilic polymer. To analyze the direct influence of the increase in the free energy of PA11 on the adhesion of the hydrophilic polymer, tests were carried out to determine the scratch resistance of the coated samples. The apparatus developed for this test was based on ASTM D 7027-05 (Standard Test Method for Evaluation of Scratch Resistance of Polymeric Coatings and Plastics Using an Instrumented Scratch Machine). Figure 1 shows a simplified diagram of this device.





Tests were performed on the samples by a conical metal tip that moved automatically at constant speed of 25 mm/min on the surface of the coated tubes. The tip was adjusted to form scratch marks around 0.1 mm. A qualitative analysis of the scratches on the surface of the samples was performed using the SEM images.

#### III. RESULTS AND DISCUSSION

Oxygen plasma, hydrogen peroxide and flame surface treatments were employed to increase wettability of polyamide 11 catheters surface. Surface hydrophilicity was evaluated through contact angle measurements and results are shown in Figures 2 a - c. The values correspond to the arithmetic means obtained in each exposure time period. The average contact angle of the untreated sample (time zero) was  $84.2^{\circ}$ .



FIGURE 2: Contact angle v 177 ersus treatment exposure time for: a) plasma; b) hydrogen peroxide; and c) flame treatments

Samples treated with hydrogen peroxide and flame treatments presented the smallest contact angles, while the oxygen plasma resulted in a less expressive reduction. An important factor, which would lead to a reduction in the contact angles, is an increase in the surface energy of the tubes after treatment. The augmentation in the electrostatic interactions between droplets and the tubes surfaces by intermolecular interactions, such as hydrogen bonds can significantly increases the wettability 15. In an attempt to achieve a further increase in the wettability of the PA11 tubes, the samples were subjected to exposure times higher than those previously established for the hydrogen peroxide and flame treatment techniques, which were 7 h and 120 s, respectively. However, the contact angle for the hydrogen peroxide (7 h) and flame (120 s) treatments were 56.5 and 47.4, respectively, indicating an increase in the contact angle for both treatments after an optimum time. This change in the surface behavior may be related to a degradation of surface. This test confirms that the exposure times established in advance for the hydrogen peroxide (4h) and flame treatments (60s) can be considered as close to ideal under this study conditions. SEM was used to evaluate the surface of samples with lowest contact angle obtained for each technique. For comparison purposes, an untreated sample was also analyzed. As demonstrated in Figure 3 a, the surface of the untreated sample showed a flat region, with only some slight scratches. Higher roughness was noted on the surface of the sample treated by plasma for 8 min (Figure 6 b), characterized by irregular gaps. Samples treated with hydrogen peroxide, and flame treatments also presented irregularities on their surface, but with the detection of micro aggregates. The increase in surface roughness of samples submitted to the proposed treatments seems to be related to the increase in the contact angles, since this feature increase the contact area between the surface and water drop deposited.



FIGURE 3: SEM images of the PA11 surfaces, magnification of 5000 times, samples: a) untreated PA11, b) treated by plasma during 8 min, c) by hydrogen peroxide during 4 h and d) by flame during 60 s.

Figures 4 a and b show the surfaces of samples treated with hydrogen peroxide for 7 h, and the flame for 120 s, respectively. Complete modification of material surface was observed in both conditions; however, hydrogen peroxide caused more pronounced changes. This characteristic corroborate with the hypothesis that after longer periods the samples surfaces started to degrade, resulting in the decrease of the contact angle. Furthermore the higher increase in surface roughness may be responsible for the increase in contact angle. A possible phenomenon that explains this behavior is the presence of micro features on the surface minimizing the contact area of the interface formed with the drop of water deposited, reducing the apparent wettability of the surface 13.



FIGURE 4: Micrographs of treated PA11 samples: a) 7 h with hydrogen peroxide, and b) 120 s with the flame.

Samples with the smaller contact angle values, as well as the untreated sample, were evaluated using FTIR. Figure 5 shows the spectra obtained at 3500 to 1200 cm-1 for the samples: untreated (Figure 5 a), treated with plasma for 8 min (Figure 5 b), hydrogen peroxide for 4 h (Figure 5 c) and the flame for 60 s (Figure 5 d).



FIGURE 5: FTIR spectra of PA11 samples: a) untreated, and treated with b) plasma for 8 min, c) hydrogen peroxide for 4 h, and d) the flame for 60 s.

Characteristic bands of the hydrophilic coating were identified for the untreated sample and for samples treated with plasma and flame16, 17. The bands at 2953 and 2923 cm-1 correspond to the asymmetric axial deformation of the C-H bonds of the CH2 groups, respectively. The band at 2854 cm-1 corresponds to the symmetric axial deformation of C-H bonds of the CH2 group. The band at 1713 cm-1 corresponds to the vibration of axial deformation of C=O. The band at 1633 cm-1 corresponds to the axial deformation vibration of C = O (amide I band) and at 1557 cm-1 to the vibration of angular deformation of NH (amide II band). The bands at 1463 cm-1 and 1377 cm-243 1 refer to the symmetric angular deformations of CH2 [17-19]. On the other hand, the sample treated with hydrogen peroxide showed spectrum characteristics similar to the PA1118,19. FTIR provides information of a material volume within some  $\mu$ m of deep penetration; therefore it is suggested that most of the coating was removed due to the chemical treatment. Untreated sample and samples treated with hydrogen peroxide (4 h) and flame (60 s), which demonstrated the best results for contact angle, were selected for the scratch test.



FIGURE 6. Micrographs of PA11 samples coated with hydrophilic film: (a) untreated PA11, and (b) treated with hydrogen peroxide during 4 h, and (c) treated by flame during 60 s.

In Figure 6 (a), the hydrophilic coating deposited on the untreated PA11 showed low adhesion to the surface, since there was a large detachment of the layer during the scratch test. Figure 6 (b) shows that sample treated with hydrogen peroxide during 4 h presents an improvement in the adhesion of the hydrophilic coating, compared with the untreated sample and small transverse cracks in the groove caused by the metal tip. This feature may be associated with the scratch of both coat and bulk materials. As observed in the FTIR results, the hydrogen peroxide treatment was observed to result in a thinner coating. On

the other hand, the sample treated with the flame for 60 s (Figure 6c) showed the best result regard to the adhesion of the hydrophilic coating. The scratch in this area is less clear, with no signs of coating detachment or tearing. This treatment was observed to result in the best emergent properties, where the coating had higher hydrophilicity and adhesion to the polymer. Future studies will evaluate the performance of this coating regard to its tribological and biocompatibility properties.

#### **IV.** CONCLUSION

The influence of exposure time for oxygen plasma, hydrogen peroxide solution and flame exposure treatments of polyamide 11 tubes was studied. It was observed that the treatment time directly affects the contact angle and there is an optimum time for each of the process investigated. SEM analysis of the surface of treated PA11 tubes allowed the observation of changes in the surface morphology and apparent roughness of the samples treated with different exposure times, and this could contribute for understand the behavior of contact angle as function of treatment time and wettability variation. FTIR results confirmed the presence of coating on samples treated with oxygen and flam as well as the thinning of the coated prepared via hydrogen peroxide. The scratch tests showed that the adhesion of the hydrophilic coating on the PA11 samples treated for 60 s with the flame presented a significant improvement over the adhesion comparing to untreated PA11 sample. The flame treatment was the most rapid and efficient process. A conclusion section must be included and should indicate clearly the advantages, limitations, and possible applications of the paper. Although a conclusion may review the main points of the paper, do not replicate the abstract as the conclusion. A conclusion might elaborate on the importance of the work or suggest applications and extensions.

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## **Thermal Insulation of Distribution Pipelines**

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**Abstract**—In heat distribution systems, which are used to supply a heat-conveying medium to consumers, a certain amount of heat is always released to the surrounding environment. Such inevitable and undesired release of heat is referred to as heat loss. Heat loss causes that the medium flowing inside the distribution system cools down and its subsequent heating up is therefore more demanding. The consequences include higher costs of heat and hot water production not only for system operators but also for end consumers. Therefore, attention should be paid to thermal insulation when incorporated in heat distribution systems, primarily to its quality and thickness.

Keywords—heat network, efficiency, heat loss, ambient temperature.

#### I. INTRODUCTION

A heat network is a pipeline system used to transport thermal energy, carried by a heat-conveying medium, from a source to an appliance. Thus, it is a system of buildings and equipment facilitating heat transport from central sources to heat consumers, regardless of their performance, nature and method of heat utilisation. A basic component of a heat network is a pipeline, including accessories.

#### II. FUNCTION OF HEAT NETWORK INSULATION

The purpose of thermal insulations of heat networks within the central supply of heat is to reduce the heat escape from a heat-conveying medium to the surrounding environment and maintain the operating temperature of the heat-conveying medium in the pipeline or a facility. Pipelines are insulated not only to prevent heat loss or cold loss, but also to prevent undesired heating [1]. If the temperature of the transported medium is higher than the ambient temperature, insulation also protects against potential burn injuries; on the other hand, at temperatures lower than the ambient temperature, it prevents pipeline misting [1].

Selection of an insulation type should be based on the temperature of the heat-conveying medium and the ambient temperature of the environment in which the heat network is to be built.

A general rule is that the appropriate insulation should exhibit minimum thermal conductivity at comparable temperature parameters; that is to say, the insulation surface temperature should be minimal in the same heat transport conditions. This is also closely related to the minimum value of the heat transfer coefficient for the heat transfer from the insulation surface to the air.

#### III. THERMAL INSULATION STATE OF THE ART

Thermal insulations are made of inorganic as well as organic materials. The key requirements that should be met by thermal insulations include: low temperature conductivity and thermal conductivity, non-absorbency, resistance to mechanical and chemical effects, they must be not aggressive to the pipeline surface and they must be hygienically acceptable.

A general rule is that the appropriate insulation should exhibit minimum thermal conductivity at comparable temperature parameters; that is to say, the insulation surface temperature should be minimal in the same heat transport conditions. This is also closely related to the minimum value of the heat transfer coefficient for the heat transfer from the insulation surface to the air.

Newly constructed or reconstructed pipelines are nowadays equipped with the pre-insulated METALNET system with the service life of as much as 30 years under stable thermal stress [3].

At the thermal stress of up to 150 °C, pre-insulated pipelines are produced as a complex system consisting of a heat pipe, insulation and a jacket pipe, together forming one compact unit. The outer surface of a heat pipe and the inner surface of the jacket pipe are connected by the insulation material which transfers forces to both pipes.

Pipelines are insulated by PUR foam consisting of freon-free polyurethane foam made of polyol and isocyanate. Pipelines may be laid underground channel-free in a HDPE jacket, or overhead in a SPIRO jacket. The HDPE jacket is made of high-density polyethylene (Fig. 1) and the SPIRO jacket is made of spiral-rolled zinc-coated steel (or aluminium) sheet (Fig. 2) [3].



FIGURE 1: Pre-insulated system for underground channelfree applications[3]



Fig. 3 shows the curves of correlations between linear thermal resistance  $R_l$  and specific heat loss  $q_l$  with an insulation radius  $r_3$  for overhead application of the pre-insulated METALNET system which exhibits a very low heat transfer coefficient ( $\lambda = 0.026 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ ). The diagram relates to the nominal diameter DN125, and the heat transfer coefficient for the heat transfer from the insulation surface to the surrounding environment was  $\alpha_2 = 3 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$ .

The graphs indicate that as the insulation thickness increased, thermal resistance  $R_l$  increased and heat loss  $q_l$  continuously decreased.



FIGURE 3: Correlation between specific heat loss  $q_i$  and linear resistance  $R_i$  with radius  $r_3$ 



Fig. 4 shows the correlation between specific heat loss  $q_i$  and insulation radius  $r_3$  for three different pipe diameters. The curves clearly show that as the inner radius of the pipe increased, specific heat loss increased with the same insulation thickness.

#### IV. ECONOMIC INSULATION THICKNESS

An insulation thickness is usually designed with the aim to achieve the highest cost saving or ensure protection of persons moving around the insulated object.

A thickness of the insulation material  $s_{ins}$  is selected depending on a type of the insulation material, required temperature of the surface to be insulated, pipeline diameter, price of the insulation material, price of heat, annual operation hours, as well as other, less important factors. The thickness that meets all the requirements above is referred to as the economic insulation thickness.

The economic insulation thickness is the thickness at which the sum of the cost of heat loss reduction and the cost of the insulation system for a given period of time reaches the lowest value. A thicker insulation reduces the heat loss and therefore also related costs, but it increases the cost of the insulation system.

The cost of insulation is not a linear function of the insulation thickness. As the insulation thickness increase, such increase in the cost of the insulation system is more intensive than the reduction of the cost of heat loss. It is always necessary to search for a compromise with the lowest possible costs. Economic insulation thickness may be identified while applying several different methods, such as the method of minimum cost of insulation [4].

The annual costs of various insulation thicknesses (annual cost of material, annual cost of installation and maintenance cost) also includes the annual cost of heat loss. The annual cost of material may be calculated as the fraction of the total cost of insulation and the planned service life of the insulation system; the same applies to the annual cost of installation. The thickness which exhibits the lowest total cost is referred to as the economic insulation thickness. The above described method of identification of the economic thickness is depicted in Fig. 5.



#### FIGURE 5: Identification of the economic insulation thickness

For a particular type of the insulation material and various thicknesses, the annual heat loss may be calculated using Formula (1); the relevant curve is the Rq Curve in the diagram.

#### 4.1 The annual heat loss is calculated using Formula [5]:

$$R_q = 3.6 \cdot 10^{-6} \cdot q_l \cdot C_e \cdot h \ (\text{\ensuremath{\in}} \, \text{m}^{-1} \cdot \text{year}^{-1})$$

$$\tag{1}$$

Wherein  $q_l$  is the specific heat loss of the pipeline (linear heat flux density) (W·m<sup>-1</sup>),  $C_e$  is the cost of energy ( $\in GJ^{-1}$ ) and h represents the operation hours per year (hour year<sup>-1</sup>).

The cost of insulation is calculated using the values of the insulation service life per annual acquisition cost; these values represent line  $R_i$  in Fig. 5.

#### 4.2 The cost of insulation calculated for one year:

$$R_i = \frac{C_i}{r} \ (\text{\pounds} \text{m}^{-1} \cdot \text{year}^{-1})$$
(2)

Wherein  $C_i$  is the total cost of the installed insulation ( $(f \cdot m^{-1})$ ) and *r* is the insulation service life (years).

The total cost, i.e. the annual procurement cost per1 m of insulation, represents the  $C_c$  Curve in Fig.5. This curve initially declines down to the minimum value, where the total cost is the lowest, and then inclines again.

#### 4.3 The total cost is calculated using the following formula:

$$C_{\rm c} = R_q + R_{\rm i} \ (\text{\ensuremath{\in}} \, \text{m}^{-1} \cdot \text{year}^{-1}) \tag{3}$$

The insulation thickness on the horizontal axis, under the minimum point, determines the economic insulation thickness for the given insulation material.

#### V. CONCLUSION

Thermal insulation represents an integral part of heat distribution systems. Its role is to prevent heat escape to the surrounding environment and maintain the required temperature of the heat-conveying medium inside the pipeline. One of the tasks of insulation materials used in industrial applications is to reduce heat loss not only in the technological equipment but also in the heat distribution system. Cost-efficiency of the operation of pipeline systems, in terms of heat and technology, is most significantly affected by a proper choice of the insulation material and a correct calculation of the thickness of the selected insulation.

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# **Dietary Assessment & Evaluation of Nutritional using Image Segmentation for Mobiles: Survey** Dr.N.Sasikala<sup>1</sup>, G.Shruthi<sup>2</sup>, E.Swapna<sup>3</sup>

Department of Electronics & Communication Engineering, Kamala Institute of Technology & Science, Singapur, Telangana Abstract— There is a great demand for easily accessible user friendly dietary self assessment and self management applications. This is because of growing concern about chronic diseases and other health problems related to cancer and obesity. So accurate diet measurement is very important. Measurement of accurate dietary and development of fully automatic estimation of nutritional intake is considered to be an open research problem. In this paper a survey on mobile telephone food record has been done. This mobile telephone food record provides an accurate account of daily food and nutrient intake of an individual. The identification and quality estimation is based on obtaining images before and after consumption of meal. Image analysis is done which includes the segmentation of food items, identification of food items by its features, automatic estimation of food portion.

Keywords— dietary assessment, diet record method, image analysis, nutrient computation, volume estimation.

#### I. **INTRODUCTION**

Chronic diseases, health problems related to diet like obesity and cancer has wide spread all over the world. Obesity is related to number of chronic diseases [13] such as type II diabetes, cholesterol, colon, breast cancer and heart disorders. The lack of balance in the eating habits is the reason for obesity. Obesity treatment is based on patient dietary intake. Dietary intake refers to the process of determining the food consumed by an individual during the course of a day. This provides valuable insights for prevention of many chronic diseases. Dietary intake measurement [19][17] is considered to be an open research problem. There should be accurate methods and tools to access the food and nutrient intake. Monitoring of nutritional status of a person is important for further clinical research. The dietary information provides the reasons for the occurrence of the disease. The dietary information also helps to use methods that prevent from the occurrence of disease.

The accurate assessment of diet is a crucial task. This task can be handled effectively if there is an availability of smart mobile phone [17] which has high resolution, imaging capability, a very fast processor, network connectivity and high memory capacity. Instead of record keepers, mobile phones [14][17] are used which provides a unique mechanism for collecting dietary information[22][17]. Dietary assessment using mobile phone improves accuracy and efficiency over personal digital assistant (PDA) with or without camera or a disposable camera.

Images are acquired before and after the consumption of food. Mobile has built in camera, by using that images are acquired, image analysis is done (like area and volume estimation before and after consumption of meal), then volume estimation [17] is done. Once the volume is estimated it is compared in the nutrient database.







Previously many works have been carried out for the automatic recognition of some types of food. A color image is taken and then it was converted into high contrast gray scale image. Intra class variance is minimized by statistical approach like global thresholding [17] of the segmented image. Small objects are removed or some areas filled by implementing morphological operations [15] like dilation and erosion in the segmented regions. For this process 5 to 6 images of the scene are utilized.

Later methods are developed which estimates the food consumption using a single image acquired from mobile device. In this method each of the food items is segmented, and is identified, then volume is estimated. Images are taken before and after meal which are used for the estimation of food intake. From this information the energy and nutrients [16][17] consumed can be determined.

#### III. IMAGE SEGMENTATION

Segmentation of the food image is done using connected component analysis active contour and normalized cuts. The amount of food in the image is measured by using a simple method. This method involves the use of a calibrated fiducial like checkerboard [17] that is placed in the field of view of the camera. With the help of this marker geometric and color correction to the images are made so as to estimate the amount of food present.

#### 3.1 Connected component labeling

There are two steps to segment food using connected components. In the first step the colour image is converted to grey scale and then thresholding [17][12] is done so that it converts into a binary image. This method is done in order to separate the food plate from the background (like table cloth). Later segmentation of food from the plate is done by eight point connected

neighbors for the low intensity value (i.e., 0) in the thresholded image (binary image). Since fixed threshold was used, pixels corresponding to the food items can be identified and the rest of the pixels can be labeled as plate.

Next the RGB image is converted in to YCbCr colour space. The mean value of histogram [17] corresponding to the plate is found by sing Cb and Cr chrominance components. Pixel locations that are not segmented during the first step are compared with the mean value of the colour space histogram of the plate in order to identify the food items. These pixels are given different label from that of the plate. Eight point connected labels for the labeled pixels are searched to segment the food item.

#### 3.2 Active contours

Techniques of curve evolution is use to detect objects by active contours method. The curve is deformed to the boundary of the object. This method is suitable for images with strong object boundaries [4][17], but are sensitive to the initialization of the active contour. Therefore region base models [18][17] are preferred which identify each region of interest by using region descriptor to guide the motion of active contour. These methods relay on the intensity homogeneity in each of the regions to be segmented and are less sensitive to the initialization of active contour. By this method partition of the image into foreground and background regions can be done. If multiple food items are connected then this model fails. Active model contour works only when the food items are separated from each other.

#### 3.3 Normalized cuts

Normalized cut is a graph partition method [6] which treats an image pixel as a node of graph and segmentation [3][17] as a graph partitioning problem. Image is modeled as a weighted, undirected graph. Each pixel is a node in the graph, and edge is formed between every pair of pixels. The measure of the similarity between the pixels is the weight of an edge[10][17].

#### **IV.** FOOD FEATURES

Color and texture are the two features that are extracted and measured for each segmented food region. Food images are taken by placing fiducial marker consisting of color checkerboard that is placed in the field view of the camera. This helps in taking the image correctly i.e. helps to correct for color imbalance in the mobile devices camera [5][17]. Color features are obtained by taking average value of pixel intensity along two color components, luminance component and two chrominance components. For texture features gabor filters [17] are used to measure local texture properties in frequency domain. Gabor filters are used for describing properties related to the local power spectrum of a signal, and this is used for texture analysis. Gabor filter is highly suitable for obtaining texture features. Each image is subjected to gabor filtering operation in a window around each pixel, and then mean is estimated along with standard derivation of energy of the filtered image

#### V. CLASSIFICATION

Classification is the process of identifying food items using statistical pattern recognition techniques based on the segmentation of food items and feature extraction. Support Vector Machines (SVM) is used for the classification [21][17] of food items. Classification involves training and testing data. Each element in training set contains one class label and several features. The features of training images are extracted and training model is generated using the SVM. The food that is labeled along with the segmented image is sent to the automatic portion estimation module where camera parameter estimation and model reconstruction are used to determine the volume of food.

#### 5.1 Volume estimation

Estimation of size of food portion from a single image is the challenging problem of image based dietary assessment. Volume estimation utilizes camera parameter estimation and mode reconstruction to determine the volume of food items. From this estimated volume the nutritional content can be determined. Volume estimation consists of camera calibration and 3D volume reconstruction [4][23] for this process two images are used as inputs. First image is the food image taken by the user and the second image is the segmented image. Later estimation of camera parameters intrinsic and extrinsic is done. Intrinsic parameters are like distortion the principal point and focal length, while the extrinsic parameters are cameras

translation [20][17] and orientation. Fiducial marker is used as a reference which poses the food. The fiducial marker is detected in the image and the pose is estimated.

The system which estimates the volume partitions the space of objects into geometric classes each with their own set of parameters. Features are extracted from the segmented region image and unprojected into 3D space. Based on the parameters of geometric class, 3D volume is reconstructed from the unprojected points. Once the volume estimation of food is made, nutrient intake consume is estimated based on USDA Food and Nutrient Database for Dietary Studies (FNDDS) [16].



**FIGURE 2: Volume estimation** 

#### VI. CONCLUSION

This is a survey paper on the Dietary assessment system using mobile devices. A measurement method was used which estimates the amount of nutrients in food image by extracting the volume of food using fiducial marker. This aids the dietitians for the treatment of obese or over weighted people or normal people so that they can have a check on eating habits. Identification of food image, image analysis, volume estimation is done which helps in building the system architecture. These techniques/methods will be useful in replacing the traditional food record methods that are currently in use. This survey is done on simple type of food. Further work/research can be carried out for multiple food items which are placed together (overlapping) on a plate. Segmentation of such food items and volume estimation is a challenging research problem.

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## Recovery of Heavy Oil from Unconventional Source (Oil Sands) with Design Steam Injection System

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**Abstract**— The use of steam ranging from 100°C to 280°C in extracting and producing oil from unconventional reservoir (oil sand) was investigated. Core samples were obtained from Edo state in Nigeria. An oil sand laboratory reservoir model was used to investigate technical feasibility of the injection of steam as an enhanced oil recovery agent for tight hydrocarbon reservoirs and the effect of temperature on the petrophysical properties such as porosity and permeability of oil sand reservoir. Steam was injected into the oil sand reservoir to recover bitumen at different temperatures ranging from 100°C to 280°C. Results showed that as the temperature of the steam increased, more hydrocarbon (bitumen) was recovered due to the reduction of its viscosity and oil-water interfacial tension. The core experiment indicates that the porosity and permeability of a tight formation such as that of oil sand at ambient temperature is 0.038 and 0.007MD respectively. An increase in reservoir. Samples collected and analyzed after recovery showed a decrease in porosity and an increase in the permeability of the formation. Results indicate that the viscosity of bitumen decreased with an increasing temperature. Maximum recovery of bitumen was obtained at 280°C. The injection of steam at temperature of 300°C and above, would produce more bitumen at industrial scale and could be refined and upgraded to liquid fuels to supplement the drastic reduction of the conventional oil. The results obtained from this work showed good agreement with data from existing data.

Keywords—Bitumen, Heavy oil, Oil sands, Permeability, Porosity, Steam, tight formation, Unconventional, viscosity.

#### I. INTRODUCTION

Oil sands are either loose sands or partially consolidated standstone containing a naturally occurring mixture of sand, clay and water, saturated with a dense and extremely viscous form of petroleum technically referred to as bitumen (or colloquially as tar due to its superficially similar appearance)(Ezra, 2011). crude bitumen is a thick, sticky form of crude oil, so heavy and viscous (thick) that will not flow unless heated or diluted with higher hydrocarbon such as light crude oil or natural gas condensate. At room temperature, it is much like cold molasses (Michael, 2009). The presence of water has long been considered to be an integral part of oil sands structure, although there is some conjuncture as to its extract nature. Early Cryotransmission election microscopy (TEM) studies provided evidence that water in natural oil sands exists as water-in-oil (w/o) emulsion (Zajic et al, 1981). On the other hand, a model of oil sands with a layer of water (approximately 10mm thick) separating bitumen from the sand surface has been proposed(Takumura, 1982). It is also necessary that the oil composition may change during thermal production of bitumen with steam, for example a common observation is that the produced oil is lighter than the original, which is believed to be a consequence of in situ upgrading processes (Montgomery et al., 2014).

Natural bitumen is oil having a viscosity greater than 10,000 centipoises under reservoir conditions and an AP1 gravity of less than  $10^{0}$  API (Hoffman, 2009). The unconventional formations may be as porous as other sedimentary reservoir rocks, their extremely small pore sizes and lack of permeability make them relatively resistant to hydrocarbons flow. The lack of permeability means that the oil and gas typically remain in the source rock unless natural or artificial fractures occur.

Unconventional oil reservoirs are characterized by unfavorable properties of rock formation, in which are accumulated, as well as unfavorable parameters of reservoir fluids. Oil reservoirs of this type occur in many places on Earth, and are the vast majority, if it comes to the global resources of hydrocarbons. In most cases, subjects to the economically justified oil production is necessary to perform the stimulation treatment, like hydraulic fracturing or acidizing. (Beckwith, 2014).Unconventional reservoirs include reservoirs such as tight-gas sands, gas sands, gas and oil shales, coal bed methane, heavy oil and tar sands, and gas hydrate deposits. The existences of these resources worldwide have been known for decades, but it was difficult and economically enviable to extract them (Milagros et al, 2018). Unconventional hydrocarbon resources are becoming a significant component of world energy consumption (Jia et al 2012; Zou, 2013). The characteristics of the unconventional resources are as follows: the sources and the reservoir coexist; the porosity and the permeability are ultra-

low; nano-scale pore throats are widely distributed; there is no obvious trap boundary; buoyancy and hydro dynamics have only a minor effect (Sun and Jia 2011; Jang et al 2013). These reservoirs require assertive recovery solutions such as stimulation treatments or steam injection, innovative solutions that must overcome economic constraints in order to make recovery from these reservoirs. The selection of the right way to drill unconventional hydrocarbon resources is closely dependent on the individual properties of the reservoir rock (Beckwith, 2014). Due to the nature of this type of reservoir the drilling process should provide access to a maximum volume of reservoir due to low permeability and heterogeneity of the rock for unconventional reservoirs is explicable to vertical horizontal and multilateral wells (stopa et al, 2015).

As the demand of finished produce of hydrocarbon increases and the abundance of unconventional oil reservoir, this work will explore recovery of heavy oil (bitumen) from unconventional source (oil sand) with designed steam injection system which will be refined to liquid fuels for our consumption.

#### II. MATERIALS AND METHOD

#### 2.1 Experimental Setup

An unconventional (oil sand) reservoir was modeled to recover bitumen using steam (at different temperature) injection. The schematic showing the design of the recovery system is presented in figure 1.



FIGURE 1: Schematic Diagram of Experimental Set Up

#### 2.2 Materials

The following materials were used for the experiment: Core samples, steam, an oil sand reservoir model, bitumen (1940g), crucible, weighing balance, tong, spatula, core holder, cement, soil water, meter rule, measuring cylinder, stop watch, thermometer, darcy flow line, beaker.

#### 2.3 Experimental Procedure

1940g of bitumen (001P) was buried in the reservoir at a depth of 1.8ft.

60 litres volume of water was pumped into the water heater through the water source and heated for  $100^{\circ}$ C.

The valves connecting the reactor (steam) and motor were opened and the steam pumped into the reservoir at a pump pressure of 0.5hp continuously while the reservoir gets heated up, melting the bitumen (turning it into a pumpable slurry) and pushing it out through the producer pump. The reservoir was evacuated and filled again with oil sands and another 1940g of bitumen at same depth and the process repeated for the temperature of 100<sup>o</sup>C, 115<sup>o</sup>C, 130<sup>o</sup>C, 145<sup>o</sup>C, 160<sup>o</sup>C, 175<sup>o</sup>C, 190<sup>o</sup>C, 205<sup>o</sup>C, 220<sup>o</sup>C, 235<sup>o</sup>C, 250<sup>o</sup>C, 265<sup>o</sup>C, and 280<sup>o</sup>C respectively and the recovery (production) made were: 45.385g, 63.08g, 80.07g, 98.04g, 116.1g, 133.8g, 151.5g, 169.2g, 186.9g, 1,904g, 1,923g and 1,939g respectively, there was no liberation of bitumen at 60<sup>o</sup>C therefore, no recovery (production) was made.

#### III. **RESULTS**

#### **3.1** Recovery at Different Temperatures

The result of the heavy oil recovered at different temperature is presented in figure 2.

The trend of the result in figure 2 shows the gradual liberation of bitumen as temperature of the steam injected was increased from 110°C to 230°C. With further increase in the temperature of the steam injected, there was a rapid corresponding increase in the (recovery) of bitumen from the sand grains. Maximum recovery was made between 230°C to 280°C. This recovery efficiency is found to be associated with minimum in oil-water intertacial tension and reduction in viscosity. Increasing the temperature of steam injected into the oil sand reservoir showed significant improvement in the recovery of bitumen oil sand rock surfaces by reducing the oil-water interfacial tension and also the viscosity of bitumen. There appeared to be a linear correlation between oil bitumen recovery rate and viscosity ratio.



#### FIGURE 2: Heavy Hydrocarbon (Bitumen) Recovery from Oil sands with Steam Injection

#### 3.2 Effect of Temperature on Viscosity and Recovery

The recovery of heavy oil as a function of temperature and viscosity is presented in figure 3. The result shows a continuous decrease of the viscosity of bitumen with increase in temperature, hence an increase in recovery as bitumen easily flows through the recovery pipe. One major hindrance in the mobility (recovery) of bitumen from deep formation is its high viscosity. The viscosity of bitumen is about one million CP  $(10^3 \text{ pars})$  at reservoir conditions. The asphatene molecule in unconventional oil (bitumen) has a high concentration of heavy polar molecules. These molecules interact strongly with the host rock oil to make the flow extremely difficult. One of the effective ways of liberating (producing) bitumen from sand grains is to reduce its intermolecular interactions by increasing the temperature to lower its viscosity.



FIGURE 3: Effect of Temperature on Viscosity and Recovery

#### **3.3** Temperature effect on porosity and recovery

The result of the influence of temperature on porosity and recovery is presented in figure 4.

From the plot, an increase in temperature gives a corresponding increase in the permeability of the formation and a decrease in its porosity.





#### 3.4 Temperature Effect on Permeability and Recovery

The relationship between the recovery and permeability with temperature is shown in Figure 5.

Permeability is high at higher temperature which corresponds to the recovery.





#### 3.5 Relationship between Petrophysical Properties, Viscosity and Temperature

Figure 6 presents the relationship between all the parameters studied at different temperature and the recovery of heavy oil.



FIGURE 6: Effect of Temperature on Petrophysical Properties and Recovery

#### IV. CONCLUSION

A technical feasibility of heavy hydrocarbon bitumen recovery in an oil sands reservoir is evaluated on the laboratory scale and from an unconventional reservoir model. The following conclusions were drawn based on preliminary findings.

The unconventional (oil sands) reservoir (sample from Edo state) is a tight formation with porosity of 0.038 and a permeability of 0.007MD.

The recovery of heavy hydrocarbon (bitumen) with the injection of steam remains challenged by factors such as low permeability and low porosity; however experimental results do indicate that the injection of steam is enhanced oil recovery method based upon encouraging increase in bitumen production obtained in the laboratory.

- The increase in the production of bitumen is dependent on an increase in temperature. As the temperature of the steam injected into the reservoir increase, recovery also increases.
- The softening and melting points temperature of bitumen are  $110^{\circ}$ C and  $116^{\circ}$ C respectively. The higher the temperature, the more bitumen softens, melts and turns into a pumpable slurry as the viscosity reduces.
- Oil recovery method based upon encouraging increase in bitumen production obtained in the laboratory.
- The increase in the production of bitumen is dependent on an increase in temperature. As the temperature of the steam injected into the reservoir increase, recovery also increases.

The softening and melting points temperature of bitumen are  $110^{\circ}$ C and  $116^{\circ}$ C respectively. The higher the temperature, the more bitumen softens, melts and turns into a pumpable slurry as the viscosity reduces.

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