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Preface

We would like to present, with great pleasure, the inaugural volume-7, Issue-10, October 2021, 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.

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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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Analysis of Personal Protection Equipment for Upper Respiratory Tract to Prevent the Spread of COVID-19 Part I

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Abstract— The number of countries where the infection by a new coronavirus has been identified is constantly increasing. Accordingly, the amounts of infected people in the individual countries are alarming. The purpose of the present article is to provide information on this new, highly infectious disease and describe potential methods for reducing the risk of the infection transmission in our environment.

Keywords— COVID-19, coronavirus, symptoms of the disease, respirator.

I. INTRODUCTION

The term coronavirus refers to four viral genera that cause diseases of various severity degrees in animals and humans. The virus may cause common difficulties, such as cold, cough and elevated body temperatures. It may also contribute to the development of deadly diseases, such as the severe acute respiratory syndrome (SARS), or support other dangerous infections.

II. COVID-19

The virus which began to spread from China in late 2019 was named 2019-nCoV (SARS-CoV-2) and it belongs to coronaviruses. Later, its name was altered to COVID-19 (on 11 February 2020). SARS-CoV-2 causes the COVID-19 disease and it was first identified in the town of Wu-Chan.



FIGURE 1: Coronavirus [1]

Covid-19 is a highly infectious disease. A median incubation period is 4–5 days, and the total reported range of the incubation period is 2–14 days. The primary transmission route is a direct contact between humans, and the disease is assumed to spread mainly via droplets during sneezing and coughing or by a body contact. There is also a risk of indirect transmission through contaminated hands, in particular by touching one's face, eyes, nose and mouth.



FIGURE 2: Coronavirus under an electron microscope [3]

This disease has symptoms similar to those of flu; that is why it is difficult to distinguish instantly between a coronavirus infection and flu. It typically affects mucous membranes of the upper and lower respiratory tracts and the conjunctiva. The most frequently reported symptoms include fever, shortness of breath, cough, fatigue and muscle pain. It may also cause the loss of smell (anosmia) and of hearing without any other signs present. In more serious cases, it may lead pneumonia, acute myocardial inflammation, organ failures and death [3].



FIGURE 3: Symptoms of the coronavirus disease 2019 [4]

In order to suppress some of the symptoms of this disease, an adjunctive therapy with commonly available medications may be initiated.

III. RISK FACTORS OF THE COURSE AND DIAGNOSTICS OF THE DISEASE

More than 80% of symptomatic COVID-19 diseases have a mild course. In approximately 15% of affected individuals, clinically serious pneumonia develops, accompanied by shortness of breath, hypoxia and extensive bilateral infiltrates visible on radiographs 24–48 hours after the disease onset. In approximately 5% of cases, intensive care is required due to respiratory distress, shock or a multiple-organ failure [5]. A critical or even fatal course of the disease may also occur in young, otherwise healthy individuals, but it is usually associated with one or two risk factors, such as cardiovascular diseases, diabetes mellitus, obesity, chronic pulmonary disease, tumorous diseases etc. Higher mortality rate of this disease is associated with a higher age of a patient.

Early and precise diagnostics of COVID-19 plays an important role in the control and mitigation of the pandemic. The most reliable method is direct virus identification by a PCR test using a sample collected from the upper and lower respiratory tract and blood serum [6].

The diagnostics may also be carried out using rapid tests, in which the virus is not identified directly in a collected sample, as it is in a PCR test; instead, rapid tests detect antibodies created by the host's immune system as an immunity response to the presence of the virus in a body.

The identification of this disease is largely contributed by knowing the travel history, especially when a person returns from a high-risk country or had been in contact with a person who was present with COVID-19.

IV. PREVENTION AGAINST COVID-19

Prevention against an infection caused by the SARS-CoV-2 coronavirus is the same as the prevention against other viral diseases, such as flu. The key measures against COVID-19 is the strict adherence to the hygiene principles, for example washing hands with soap and disinfection, the use of facial protective equipment (face masks, respirators, protective masks and shields etc.) when interacting with other people, and social distancing. Other efficient measures against this disease include avoiding touching one's eyes, coughing and sneezing into a paper tissue, and avoiding journeys to high-risk countries.

A face mask acts as a mechanical barrier that prevents saliva and phlegm droplets or aerosols from penetrating out of the respiratory tract. The total service life of a disposable surgical face mask is approximately 3 hours.



FIGURE 4: Disposable face masks

A respirator protects a healthy individual against viruses from the surrounding environment. It filtrates the air inhaled by a wearer and traps harmful substances to prevent them from penetrating the respiratory tract. The face masks which are recommended for the protection against viruses are FFP2 or FFP3.





Disposable respirator FFP2 NR Disposable respirator FFP3 NR FIGURE 5: Disposable respirators

A one-way exhalation valve makes breathing much easier. If a wearer is ill and sneezes, the valve lets the sneezed out droplets into the surrounding air, and this imposes a risk for the persons around. Therefore, these respirators are suitable mainly for the healthy front-line staff (healthcare professionals, police officers, fire-fighters).



Disposable respirator FFP2 Disposable respirator FFP3 FIGURE 6: Disposable respirators with a one-way inhalation valve

The staff providing prolonged healthcare are advised to use FFP3 respirators with replaceable filters, which exhibit lower resistance during breathing, but still maintain their filtration capacity (Fig. 7).



FIGURE 7: Reusable respirator with replaceable filters [7]

The Table below presents the efficiency data for the individual types of the personal protection equipment, which are recommended by the WHO.

EFFICIENCE OF THE PERSONAL PROTECTION EQUIPMENT USED FOR THE FACE [6]						
Equipment type	Cleth feas mode		Respirator			
	Cloth face mask	Surgical face mask	FFP1	FFP2	FFP3	
Efficiency (%)	30–60	50-80	80	94	99	

 TABLE 1

 Efficiency of the personal protection equipment used for the face [8]

Even though cloth or surgical face masks do not offer a hundred-percent efficiency of the protection against infections, wearing them is reasonable as it mitigates and minimises the spread of viruses. Protection efficiency may be increased by using these face masks in combination with protective shields or goggles and disposable rubber gloves.

V. CONCLUSION

The number of persons infected with coronavirus is constantly rising and this fact should not be underestimated. The infection may be transmitted as easy as just by being near a person who is present with symptoms like cough, fever or difficulties breathing, as well as by touching objects that such a person has recently touched. Despite the fact that efficient vaccines against COVID-19 have been developed, it is necessary to consistently implement the key measures that eliminate the transmission of this highly infectious disease.

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Analysis of Personal Protection Equipment for Upper Respiratory Tract to Prevent the Spread of COVID-19 Part II

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Abstract— The coronavirus pandemic significantly affects the lives of families, employees, employers, or sole proprietors, and it is therefore necessary to prevent its spread by using appropriate protective equipment. The article discusses the possibilities of protection against various forms of harmful particles, including the highly infectious virus SARS-CoV-2 using face protection respectively respiratory protection.

Keywords— COVID-19, respiratory protective equipment, surgical face masks.

I. INTRODUCTION

Clinicians and experts in the field of public health recommend preventive measures, including use of respiratory protective devices, to reduce the disease caused by infection with severe acute respiratory syndrome coronavirus 2 (known as SARS-CoV-2). It is thought that SARS-CoV-2 is spread primarily through contact and large respiratory droplets, but evidence also indicates potential transmission by fine respiratory aerosols located in the surrounding environment. [1 - 3] There are available several types of face masks and the respiratory protective equipment to be used in hazardous work environments, including environments contaminated by a new virus SARS-CoV-2.

II. ANALYSIS OF HARMFUL COMPONENTS OF THE ENVIRONMENT AFFECTING THE RESPIRATORY SYSTEM

In the environment, we may encounter components that negatively affect our breathing and our overall health. These are components such as dust, smoke, microorganisms, allergens (pollen, mold, etc.), solid pollutants caused by anthropogenic activity or the influence of nature (Fig. 1).





Small, dry, solid particles can be projected into the air by natural forces, such as wind, volcanic eruption, and by mechanical or manmade processes such as crushing, grinding, milling, drilling, demolition, shoveling, conveying, screening, bagging, and sweeping. Dust particles are usually in the size range from about 1 to 100 µm in diameter, and they settle slowly under the influence of gravity. [5]

Other pollutants may include vapors generated by the cooling of molten metal. The hot material reacts with oxygen to form oxides. When melting lead e.g. lead oxide fumes are formed; welding produces iron oxide and other metal fumes.

When coal is burned, smoke is produced. Smoke is made up of small particles of coal and soot, which combine with droplets and solid particles.

Particulate matter (PM) is the air pollutant that causes the greatest harm to human health in Europe. They are so light that they can float in the air. Some of these particles are so small (one thirtieth to one fifth of the diameter of a human hair) that not only do they penetrate deep in our lungs, but they also pass into our bloodstream, just like oxygen. Some particles are emitted directly into the atmosphere. Others come about as a result of chemical reactions involving precursor gases, namely sulphur dioxide, nitrogen oxides, ammonia, and volatile organic compounds. [5]

Microorganisms that spread in the environment include bacteria, viruses, spores and the like. Currently, the most resonant among the viruses is SARS-CoV-2 (Severe Acute Respiratory Syndrome Coronavirus 2, formerly temporarily known as 2019-nCoV), which belongs to the genus Betacoronavirus and was first identified in patients with severe respiratory disease in December in 2019 in the Chinese city of Wuhan. [6]

COVID-19 (Corona Virus Disease 2019) is manifested by flu-like symptoms. Therefore, the disease was initially compared to influenza and downplayed. These symptoms may vary in intensity in different people. It depends mainly on the strength of their own immunity. Therefore, the disease is most dangerous in the elderly and people with associated health problems.

III. ANALYSIS OF HARMFUL COMPONENTS OF THE ENVIRONMENT AFFECTING THE RESPIRATORY SYSTEM

There are several types of face masks and respiratory protection. The choice of filter device or breathing apparatus depends on criteria such as [7]:

- Oxygen content,
- > Type and concentration of contamination,
- ➢ User mobility,
- ➢ Required level of protection.

Respiratory protection equipment can be divided into two main groups (Fig. 2) [7]:



FIGURE 2: Respiratory protection

3.1 Filtering Respirators:

Its depend on ambient air.

The air is cleaned when drawn through the filter. Respirators are not suitable for use in the IDLH (Immediately Dangerous to Life or Health) environments including oxygen-deficient atmospheres.

Subdivision in this group:

- > Negative pressure filtering devices for instance disposable-, half- and full face masks with filters,
- > Positive pressure filtering devices for instance powered air hoods and masks.

It should be added that respirators can also be equipped with exhalation valves.

3.2 Respiratory Devices (DA)

These are independent of ambient air.

Breathing air is supplied from an external source, e.g. compressed air or pressure cylinders. The user is connected to a hose or uses an independent breathing apparatus. It is less mobile due to hose or time constraints. The respirator is suitable in environments where there is an immediately dangerous to life or health and in environments with a lack of oxygen (unlike filter respirators).

The group of the respiratory devices consists of:

- Compressed air treatment plant,
- Independent breathing apparatus.

Respiratory protection masks protect the lungs from the ingress of dust, smoke and liquid mist (aerosol), but do not protect against steam and gas. The classification system is divided into three classes of FFP (filtering face piece).

The respirator covers the nose and mouth and consists of various filter materials and the mask body itself. Their wearing is prescribed at workplaces where the highest exposure value at work is exceeded. This is the maximum permissible concentration of dust, smoke and aerosols in the breathed air, which does not lead to damage to health. If it is exceeded, the obligation to use respiratory protection masks applies.

The EN 149 standard [8] defines three classes of filter efficiency, namely FFP1, FFP2 and FFP3. Before we move on to the FFP classes, it is useful to understand the following terms:

- Occupational Exposure Limit (OEL) OELs are standards that determine the amount, or concentration, of a hazardous substance allowable in the workplace air.
- Assigned Protection Factor (APF) The APF of a mask indicates how well it protects the wearer from hazardous substances. A safety mask with an APF of 4 will reduce the concentration of the hazardous substance in the air that is breathed by the wearer by 4 times.

It is possible to protect the respiratory tract from dust particles and aerosols with respirators as well as filters of the following protection classes (Table 1 – Table 3) [9]:

TABLE 1CHARACTERISTICS OF FFP1

	 Protection against non-toxic dust and dust which does not cause fibrosis, Inhelation does not load to disease but may invitate the requirement tract and the substances may small
	<i>unpleasant,</i>
I	> The total leakage rate may not exceed 25%,
TFP	> This kind of mask may be applied under a fourfold OEL transgression at the most.
I	Respiratory masks with protection class FFP1 are suitable for use in work environments where toxic or fibrogenic dusts and aerosols are not expected. They filter at least 80% of the particles in the air up to 0.6 μ m in size. The use of FFP1 respiratory protection masks is usually sufficient in the construction and food industries.

TABLE 2CHARACTERISTICS OF FFP2

- Protection from firm and fluid deleterious kinds of dust, smoke, and aerosols,
- Particles may be fibrogenic which means they irritate the respiratory system in the short term and can result in reduction of elasticity of pulmonary tissue in the long run,
- Total leakage may amount to a maximum of 11%,
- > OEL transgression to the tenfold value.

FFP2

FFP3

Protection class FFP2 respirator masks are suitable for use in work environments with the presence of harmful and mutagenic particles can be found in the breathing air. Respirator masks of this class must capture at least 94 % of the particles measuring up to 0.6 µm and may be used in environments transgressing the OEL up to a maximum of the tenfold concentration. The same goes for the TRC value (technical reference concentration). Protection class FFP2 respirator masks are often worn in the metal and mining industry. In these environments, workers come into contact with aerosols, mists and smoke, which in the long run lead to respiratory diseases, such as lung cancer, and significantly increase the risk of subsequent diseases, such as active pulmonary tuberculosis.



TABLE 3CHARACTERISTICS OF FFP3

- Protection from poisonous and deleterious kinds of dust, smoke, and aerosols,
- > When working with oncogenic or radioactive substances or pathogens such as viruses, bacteria and fungal spores FFP3-class respirator masks are recommended,
- ▶ Total leakage may amount to a maximum of 5%,
- > OEL transgression to the thirtyfold value.

Protection class FFP3 respirator masks offer maximum protection from breathing air pollution. The total leakage may amount to a maximum of 5% and they must filter 99% of all particles measuring up to 0.6 μ m. This kind of mask also filters poisonous, oncogenic, and radioactive particles. Protection class FFP3 masks are used in working environments transgressing the OEL by the thirtyfold industry-specific values. They are often used in the chemistry industry.



As far as face masks are concerned, a wide range of half masks (a), full-face protective masks (b) as well as comfortable and adaptable respirators with a soft thermoplastic seal (c) have been developed (Fig. 3).



FIGURE 3 Types of face masks and replaceable filters [7]

IV. REQUIREMENTS FOR RESPIRATORS EFFECTIVE TO PROTECT AGAINST COVID-19

The current epidemiological situation results in an increased demand for respirators, which are classified according to their filtration efficiency and maximum overall penetration into the FFP3 class (protection against organic, inorganic particles and biological particles such as viruses, bacteria, fungi, etc.) [10].

However, respirators with invalid or false certificates may also be on the market in this context. These are respirators that are imported from third countries. Notices of these certificates (issued by a person not notified for their assessment) are also on the European website of the European Safety Federation.

Before importing or purchasing respirators of category FFP3, it is necessary to check whether the given PPE has [11]:

- EU Declaration of Conformity (the 4-digit number of the notified body must be the same as the 4-digit number of the notified body after the CE marking),
- > CE marking + 4-digit number of the notified body directly on the FFP3 respirator.

Applicable legal frame of the respiratory protection in the European Union is given in the Tab. 4.

	Protective mask*	Medical mask*
Other terms used	FFP2 / FFP3 / FFP1 / respirator / filtering face piece / filtering half mask	Surgical mask
Function	Protect the wearer from harmful dusts or aerosols / particles in the air	Protect the patient by reducing the risk of spreading infective agents via exhaled air of the wearer
Applicable legal frame	PPE Regulation (EU) 2016/425 – category III	Medical Devices Directive 93/42/EEC – class I (class IIa for sterile medical masks)
Marking	CE + 4digit identification number of the notified body responsible for the production follow-up	CE
Applicable EN standard	EN 149	EN 14683

TABLE 4 APPLICABLE LEGAL FRAME OF THE RESPIRATORY PROTECTION [12]

*: Some masks are both Protective and Medical mask and comply to both legal acts and are tested to all relevant standards for both functions.

Labelling of the respirators in the US and in Europe is different. In the USA, it is recommended to use N95 particulate respirators certified by the National Institute for Occupational Safety and Health. The N95 series respirators are made of a material that has 95% particle filtration efficiency of the particles having a diameter of $0,3\mu$ m. In Europe, respirators marked FFP2 (= N95) or FFP3 (= N100) are comparable.

V. USING OF PPE IN HEALTHCARE FACILITIES AS A PREVENTION OF THE SPREAD OF COVID-19

Centres for Disease Control and Prevention and the World Health Organization (WHO) recommend respirator use especially for the following workers [13]:

- > Health care workers entering the room of a patient with COVID-19,
- > Medical transport workers transporting patients with suspected COVID-19.

A face mask or respirator without an exhalation valve is used to capture infectious droplets from the patient's airways. In the case of respirators with an exhalation valve, the exhaled air from the patient must pass through an additional filter layer which traps infectious substances e.g., by applying a surgical mask to a respirator with an exhalation valve.

Respirators are not routinely needed (by staff or visitors) in other parts of hospitals or other health care facilities where there is no direct contact with patients. [13] Most people who develop disease COVID-19 become infected by contact with other

people who are ill or by breathing contaminated air. Since the beginning of the pandemic, there has been an increased emphasis on surface disinfection as a possible source of coronavirus infection. Experiments aimed at surviving the virus on surfaces show that the coronavirus can survive on surfaces for longer, but that does not mean that people catch it from surfaces such as door handles. Most studies on the survival of viruses on various surfaces have been performed under laboratory conditions. Only a few studies looked for a viable virus outside the laboratory. The results of these experiments showed that none of the viral materials were able to infect the cells. [14 - 21]

The best protection against the virus not only in medical facilities but also in public places with a higher concentration of people at a time of increased incidence of respiratory diseases is the wearing of face masks. As secondary protection especially after taking a face mask it is recommended washing or disinfecting hands and not to touch facial mucous membranes (eyes, mouth, or nose).

All healthcare professionals should evaluate the infection risk and wear appropriate personal protective equipment (PPE) to minimize this risk before each interaction with a patient suspected or confirmed of COVID-19 [22].

The following table (Tab. 5) evaluates the need to use the surgical face mask or the FFP3 respirator for healthcare professionals.

USING SURGICAL FACE MASK VS. FF15 RESI IRATOR [25]				
When to us	se a surgical face mask	When to use an FFP3 respirator		
In cohorted area (butno patient contact)	Close patient contact (within one meter)	Carrying out potentially i generating procedures	infectious aerosol	
For example: Cleaning the room, equipment cleaning, discharge patient room cleaning, etc.	For example: Providing patient care, direct home care visit, diagnostic imaging, phlebotomy services, physiotherapy, etc.	For example: bronchoscopy, endotrach intubation, tracheostomy cardiopulmonary resuscit diagnostic sputum induct	eal procedures, tation, ion:	
PPE to be worn:	PPE to be worn:	 Where a patient is kn an infection spread v When caring for patient to be infected with a infectious respiratory 	own/suspected to have ia the aerosol route ents known/suspected newly identified v virus	
Surgical face mask (along with other designated PPE for cleaning)	Surgical face mask Apron Gloves Eye protection	PPE to be worn: FFP3 respirator Gown	Gloves Eye protection	

 TABLE 5

 USING SURGICAL FACE MASK VS. FFP3 RESPIRATOR [23]

VI. CONCLUSION

Based on previous experience and knowledge, SARS-CoV-2 has been shown to spread primarily through close personal contact with symptomatic individuals and also through contaminated surfaces with infectious droplets and subsequent transmission of these droplets to the mucous membranes of the eyes, nose or mouth. It is assumed that the transmission of the virus occurs through the very small air particles.

At present, several PPE are available on the market. PPE is used to protect health as well as the respiratory tract in the work environment. These devices have also found application in reducing the risk of transmitting COVID-19, especially in the

front-line sectors. In an effort to eliminate this infection, a number of countries have engaged in research of the new materials for the production of filters as well as the types and shapes of protective face masks.

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The Effects of Pipeline Parameters on Dispersion Process of Odourant in Natural Gas Mixture

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Abstract— This article discusses the possibility of natural gas odourisation, which is performed due to the fact that natural gas has no smell of its own. In order to facilitate the detection of potential gas leaks from distribution networks by the human sense of smell, the gas must be combined with an odourant. A concentration of such an odourant should be constant throughout the entire distribution network in order to facilitate the earliest possible detection of potential gas leaks.

This article also deals with the issues related to the odourisation of natural gas in a high-pressure pipeline in a gas distribution network. It presents a theoretical overview of the used types of odourants, odourant control methods, and a model of odourant behaviour in a distribution network in various conditions, which was created using numerical simulations performed in ANSYS software.

Keywords—natural gas, odourisation, ANSYS, pipeline.

I. INTRODUCTION

Natural gas is an important fossil fuel, which is used in multiple industries. When compared to other fossil fuels, the amount of harmful substances produced during the combustion of natural gas is smaller; moreover, natural gas does not produce any mechanical impurities, such as carbon black. Therefore, it may be classified as an ecological fuel. Due to the fact that it is odourless and lighter than the air, it is regarded as a problematic material in terms of safety. As a result, the detection of potential leaks of natural gas from a distribution network is conditioned by the presence of an odourant in a mixture.

II. NATURAL GAS ODOURISATION

Odourisation means the addition of substances with strong scents to natural gas. The odourisation process belongs to the fundamental processes in every company that operates in the field of gas transportation. Precise addition of an odourant, appropriate monitoring techniques and maintenance represent important factors that affect the development of an effective odourisation process. The importance of odourisation consists in providing an early warning for people in the event of an accidental gas leak from a gas pipeline or from an end-user gas facility. A choice of a correct odourant depends on a particular area of application and is affected by a variety of safety-related and economic factors. The odourants which are typically used in natural gas odourisation include sulphur-containing odourants (mercaptans, sulphides, and heterocycles containing sulphur).

III. ODOURANT CONTROL IN A MIXTURE WITH NATURAL GAS

A concentration of an odourant in natural gas changes due to the effects of various physical and chemical factors. However, in order to ensure safety, the gas must be treated so that it acquires a required level of scent at each point of a distribution network. The presence of an odourant in natural gas must be controlled on the regular basis. At present, a concentration of an odourant is usually tested by olfactometry, in which a smell intensity of the odourised gas is assessed by a human sense of smell. This method belongs to subjective control methods. Objective methods for the control of an odourant in a mixture with

natural gas include chromatography. It is a laboratory separation method, which is used for the precise identification of an odourant concentration in natural gas. The underlying principle of chromatography is the separation of the individual mixture components between the mobile and the stationary phases based on different velocities of the individual mixture components. The presence of an odourant is identified using also electrochemical sensors that use electrodes placed in a gel electrolyte. The electrolyte and electrodes are separated from the surrounding atmosphere using a diffusion barrier, through which molecules of the measured gas penetrate and subsequently react with the electrolyte. An oxidation-reduction reaction on the electrodes results in a change in the cell potential. A rising potential means that a gas concentration increases.

IV. THE PROCESS OF ODOURANT DISPERSION IN NATURAL GAS IN THE CFD SOFTWARE ANSYS CFX

A model of odourant dispersion in a high-pressure pipeline was created using numerical modelling, which may be used in addition to, or instead of, traditional experimental procedures. The modelling consists of designing a model, performing a simulation, and analysing the obtained data. The model designing step includes the determination of the initial and boundary conditions and the creation of the model itself. During the simulation, it is important to visualise and observe the problem. The advantages of a numerical simulation include a possibility of verifying several problem solution alternatives without reaching a high-cost level; if the adjustments are made after the device is already manufactured, the costs are high. The advantages also include the possibility to simulate the unacceptable long process in shorter duration.

The ANSYS CFX software environment uses the Finite Volume Method (FVM), which is based on the principle of representing differential equations in a form of a system of differential equations. The solution accuracy depends on a selected mesh density and on a differential scheme. It can be expected that as the finite volume mesh becomes finer, the more precise the problem solution will become.

V. THE EFFECT OF A PIPELINE LENGTH ON ODOURANT DISPERSION IN A MIXTURE WITH NATURAL GAS

For the purpose of investigating the effects of different pipeline lengths on odourant dispersion in a mixture, a simulation was carried out with two pipes of different lengths -3 m and 30 m, but of identical diameters (D = 200 mm). The input parameters, i.e. the odourant concentration, natural gas flow rate and the pressure inside the pipeline, were identical for both types of pipe. The simulation results were evaluated by comparing the changes in concentration along the lengths and cross-sections of both pipes with different lengths.



FIGURE 1: Odourant concentration in a mixture with natural gas at a cross-section at the pipe outlet

The changes in odourant concentration in a mixture with natural gas along the entire length of the investigated pipes are shown in Fig. 2 and Fig. 3; they show that the odourant concentration did not exhibit any changes throughout the entire investigated section of the pipeline.







FIGURE 3: Changes in concentration along the length inside a 30 m pipe

Based on these findings, it is possible to state that with identical input parameters, i.e. the mass flow rate, pressure and the odourant concentration, the length of the pipe has no effect on changes in the odourant concentration in a mixture with natural gas.

VI. THE EFFECT OF PIPELINE ELEVATION ON ODOURANT DISPERSION IN A MIXTURE WITH NATURAL GAS

The investigation into the effects of pipeline elevation was carried out using two predefined pipeline sections, similar to those in the previous case. In these sections, changes in the odourant concentrations resulting from by different elevation values were compared, while the input parameters remained unchanged. In the first case, the flow in the main pipe with a diameter D = 200 mm was monitored. A delivery pipe with a diameter D = 80 mm was attached to this pipe at an angle of 45° and 5 m below the level of the main pipe (Fig. 4). In the second case, the pipes used were of identical diameters, but the delivery point was 5 m above the level of the main pipe.







FIGURE 5: Changes in concentration with a descending elevation of -5 m

Fig. 5 indicates that a lower attitude of the investigated pipeline point correlates to a higher odourant concentration. However, it must be noted that in descending elevation of -5 m, an odourant volume fraction only begins to change at 10th decimal place. Such changes in concentration are practically immeasurable, and hence negligible. The same result may be observed with ascending elevation (Fig. 6). Despite the fact that the odourant concentration in the mixture decreases as the altitude of the delivery point increases, such a change occurs again at the 10th decimal place; this means that the change is negligible.



FIGURE 6: Changes in concentration with ascending elevation of 5 m

Based on a comparison of the results of both simulations, it is possible to state that pipeline elevation, similarly to a pipeline length, has no significant effect on changes in a concentration of an odourant contained in natural gas.

VII. CONCLUSION

The addition of odourants to natural gas is an important process, as it facilitates warning people in the event of an accidental gas leak from a gas distribution pipeline or from an end-user gas facility, and hence it prevents the risk of fire, explosion and intoxication. In our investigation, dispersion of an odourant in a natural gas distribution network was homogeneous, and the odourant together with natural gas formed a homogenous mixture, which remained unchanged in practically all sections of the pipeline. The pipeline inclination and length had no significant effects on changes in the odourant concentration.

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Design of Heat Exchanger for Low-Pressure Vessel for Hydrogen Storage

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Abstract— This article describes the issue of hydrogen storage in metal hydrides and addresses the design of a heat exchanger and the calculation of heat transfer in low-pressure steel vessel with heat exchanger, which is used for hydrogen storage using La Ce Ni based metal alloys.

Keywords—Heat exchanger, heat transfer, Hydrogen, hydrogen storage, metalhydride vessel.

I. INTRODUCTION

Following the problem of global warming, efforts have intensified to explore and implement alternative energy sources. At present, various political parties around the world are trying to raise awareness of renewable energy sources. In this context, hydrogen is considered a suitable candidate precisely because of its high heat value and good effects on environment. However, hydrogen is an energy carrier rather than source of energy, so it requires an efficient energy storage mechanism. There are several ways of storing the hydrogen, such as: high-pressure gas storage, liquid cryogenic storage, and low-pressure storage in metallic alloys. Low-pressure vessels are currently being developed for hydrogen storage. The main obstacle of the low-pressure vessels is the need to cool the alloys during hydrogen absorption and to heat the alloys during desorption.

II. DESIGN OF THE LOW-PRESSURE VESSEL

The design of the pressure vessel must be based by standard STN EN 13322-2. This standard is about transport gas cylinders, design and production of refillable steel gas cylinders. The construction of vessel consist of main body and casing for the flow of coolant and heating fluid as can be seen in Fig 1.



FIGURE 1: Design of low-pressure vessel

Stainless steel 1.4404 316L with parameters shown in Table 1 was chosen for the construction of the vessel.

TABLE 1						
MECHANICAL PROPERTIES OF STAINLESS STEEL 1.4404 316L						
0,2% Re (MPa)	Rm (MPa)	ρ (kg·m ⁻³)	μ	E(MPa)		
200	500-700	8000	0.3	$2.1 \cdot 10^5$		

Where Re- yield strength, Rm- tensile strength, p- density, µ- Poisson's constant and E- Young's modulus of elasticity

III. DESIGN OF THE HEAT EXCHANGER

The main task of this work is to design an internal heat exchanger to improve heat transport during the filling of the pressure vessel with hydrogen. The geometry design of the heat exchanger consists of five main fins and ten secondary fins. The secondary fins are connected to the main fins with circular section which copies the shell of the pressure vessel. This should increase the heat exchange area at the wall of pressure vessel. The gap between the heat exchanger and the pressure vessel is 1mm. The heat exchanger is shown in Fig. 2.



FIGURE 2: Cross-section geometry on heat exchanger

IV. SETTING THE SIMULATION FOR HEAT EXCHANGER

In this simulation we, will consider a heat exchanger that consists of ten main fins and ten secondary fins as shown on Fig. 2. The first step in setting up the simulation is to create a simulation model in Ansys Workbench. The simulation model consists of three main parts which are the metal hydride, heat exchanger and shell of pressure vessel. After creating each part of the model, it is necessary to generate a mesh for each part of the model as shown on Fig. 3. The next step of simulation is to define boundary conditions. During filling, the pressure vessel is cooled by water on its outer surface. It is assumed that the temperature of the water is approximately 20°C and flows at a speed of $0.3 \text{m} \cdot \text{s}^{-1}$. To simplify the simulation, we calculated the heat transfer coefficient between the pressure vessel and cooling water and thus obtained how much heat we can dissipate by water cooling, which we used as a boundary condition for heat dissipation to the environment. Subsequently, we defined a metal hydride material (La_{0.85} Ce_{0.15} Ni₅), which has the following properties:

- Molar mass: $62,55 \text{ kg} \cdot \text{kmol}^{-1}$,
- > Bulk density: $3250 \text{ kg} \cdot \text{m}^3$,
- > Specific heat capacity: 430 J.kg⁻¹ · K⁻¹,
- > Thermal conductivity: $1 \text{ W.m}^{-1} \cdot \text{K}^{-1}$.

The secondary fins are connected to main fins near the wall of pressure vessel. In the simulation, filling time 1200s is considered.



FIGURE 3: Generated mesh of the model for simulation

 TABLE 2

 SELECTED VALUES OF SIMULATION PARAMETERS TO CALCULATE INTERNAL SOURCE

Volume of metal hydride	15 698 mm ³
Mass of metal hydride	$51,0185 \cdot 10^{-3} \text{ kg}$
Mass of stored hydrogen	$7,295 \cdot 10^{-4}$ kg,
Volume of hydrogen	$8,117 \cdot 10^{-3} \mathrm{m}^3$
Generated heat	8 198,26 J
Internal generated power	6,8318 W
Intensity of the internal heat source	$435,2 \text{ kW} \cdot \text{m}^{-3}$

In Fig. 4 we can see the design after defining all domains and boundary conditions as well as intensity of the internal heat source.



FIGURE 4: Boundary conditions of the simulation

V. RESULTS OF SIMULATION

The temperature field in cross-section view of the pressure vessel is shown in Fig. 5. The maximum temperature in pressure vessel after 1200s is 89.53°C. The warmest places in metal hydride are located between the secondary fins. The heat generated is well dissipated from the vessels core.



FIGURE 5: Temperature field of pressure vessel with heat exchanger

Fig. 6 shows the maximum and minimum temperatures over a period of 1200s. On the maximum temperature curve, we can see that during the last 200 seconds of the simulation it is almost stable. The minimum temperature curve does not change from the half of the simulation and remains at the same value till the end of the simulation. On Fig. 6 is also shown the course of the subtraction between the minimum and maximum temperatures (ΔT).



FIGURE 6: Courses of maximum and minimum temperatures in pressure vessel and course of ΔT





Fig. 7 shows the heat dissipation from the pressure vessel by cooling around the circumference of the steel shell. The highest values of heat dissipation are in the places where the fins are located, whether primary or secondary.



FIGURE 8: Cooling performance over time

Fig. 8 shows the water-cooling performance of the pressure vessel over the total filling time. The maximum value peaks at 6.81W per 1 mm of model thickness that was reached at the end of the simulation. We can observe that the curve stabilizes at the end of the simulation and does not have an increasing tendency.

VI. CONCLUSION

Hydrogen technologies represent a promising future in the automotive industry but also in everyday use. Hydrogen has a low bulk density and therefore must be stored in high-pressure tanks or in liquid form using cryogenic vessels, which makes these methods in terms of energy efficiency very demanding. From this point of view, the storage in metal hydrides may represent a promising future. When hydrogen is stored in metal hydrides, high pressures are not required, and they reach high values of bulk density. The disadvantages of storage in metal hydrides are the low bulk density in metal structure and the very low value of thermal conductivity of the used alloys. The aim of this work was to design a heat exchanger that will efficiently dissipate heat from the core of the pressure vessel to the outer jacket, where the vessel is cooled by coolant. The results of the simulation showed that the design of the heat exchanger achieved very satisfactory results and values in all aspects of the investigation.

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New Phytochemical Profile by GC-MS of Toluene Extract of Rhizomes from *Smilax domingensis* in Cuba

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Abstract— A preliminary chemical characterization of main components of toluene extract with dried rhizomes of Smilax domingensis Willd. that grow in Cuba was done using a GCMS-QP2010 Ultra Shimadzu and the mass spectra of the compounds found in the extract was matched with the National Institute of Standards and Technology (NIST) libraries. After sample derivatization 893 chemical compounds were registered by the equipment and from them, 30 different chemical components were characterized and reported for the first time from this part of the plant in our country. The results demonstrate that the developed method could be employed as a rapid and versatile analytical technique for identification of chemical constituents and quality control of Smilax domingensis.

Keywords— Smilax domingensis, chemical constituents, GC-MS, rhizomes, toluene extract.

I. INTRODUCTION

Smilax domingensis Willd, Smilacaceae, known as bejuco chino or raíz de china, zarzaparrilla de la tierra (Cuba); bejuco de membrillo, dunguez blanco (Puerto Rico); chiquihuite (México), is a climbing shrub from Tropical America. The rhizome is popularly used in medicine as anti-inflammatory, antiseptic, depurative, sudorific, anti-asthmatic, anti-herpetic, anti-rheumatic and for venereal diseases (Roig, 2014).

Smilacaceae is a family of climbing shrubs represented by the single genus Smilax with close to 250 species worldwide, present with 26 species in Mesoamerica (Huft, 1994). Widely used since ancient times, the main species reported are *Smilax aristolochiaefolia* Mill., *S. febrifuga* Kunth, *S. ornata* Hook, and *S. regelii* Killip & Morton, characterized by roots and small rhizomes used as antiseptic and anti-pruritic drug (British Herbal Pharmacopoeia, 1983).

Smilax domingensis Willd. is native from Tropical America, growing in lowlands, in humid forests of wide-leaved species (Standley & Steyermark, 1952). Although widely used, there are several taxonomic difficulties. Few anatomic studies of American Smilax have been carried out, particularly for species from Argentina (Guaglianone & Gattuso, 1991) and Brazil (Andreata, 1997).

In the scientific literature, there are some data of the phytochemical components and pharmacological actions while a small number of data of standards for identification and authentication about *Smilax domingensis* Willd. In Cuba, there is not available information for this spice. The main components found and shared by most species of the genus are the steroidal saponins, phytosterols, and triterpenoids (British Herbal Pharmacopoeia, 1983).

It is an evergreen dioic woody vine, 2-4 m high with lignified rhizomes. Rhizome is voluminous, with tuberous swelling, reddish brown in color, measuring 14-21 cm long, 3.925 cm wide and 3.175 cm high. The average weight is around 87.05 g. Roots are adventitious, growing from the rhizomes (Figure 1).



FIGURE 1: Macroscopical view of rhizomes from *Smilax domingensis* Willd. in Cuba.

Between 2017 and 2018, a Cuban research team reported a preliminary study about the pharmacognostic, physicochemical and phytochemical characteristics from this part of the plant, showing the values of moisture content (13.11%), extractable matter in ethanol at 70% (13.53%), total ashes (3.45%), water soluble ashes (2.43%) and acid insoluble ashes (0.64%). Phytochemical screening revealed the possible presence of alkaloids, oils and/or fats, coumarins, saponins, flavonoids, pyrogallol-type tannins, quinones, catechins, reductants sugars, triterpens and steroids and absence of resins, aminoacids, cardiotonic glycosides, anthocyanidins and astringent and/or bitter principles, realized under WHO parameters (Yaque, J.G. et al., 2017).

A preliminary chemical characterization of main components of ethanolic extract at 95 % with dried rhizomes of *Smilax domingensis* Willd. that grow in Cuba was done using a GCMS-QP2010 Ultra Shimadzu and the mass spectra of the compounds found in the extract was matched with the National Institute of Standards and Technology (NIST) library. 35 different chemical components were characterized and reported for the first time from this part of the plant in our country, most of the chemical components belong to organic acids, reductants sugars, and lactones and relative compounds. The chemical matches were around 80 % of coincidence with NIST21 and NIST107 libraries (Soledispa et al., 2018).

The aim of this research was to characterize the chemical composition of rhizomes of *S. domingensis* from our country for the development and utilization of the promising medicinal plant.

II. MATERIALS AND METHODS

2.1 Plant Material and Reagents

The S. domingensis Willd. rhizome was collected from Sierra Cristal, Sagua de Tánamo, Holguín Province, Cuba, 850-1000 masl, by Elio M. García Fargie in March, 2016. The Voucher No. HAJB 089193 was registered at National Botany Garden in Havana, Cuba. The plant material was authenticated by Dr. Jorge E. Gutiérrez Amaro. The harvested rhizomes were dried in the shade at room temperature (temperature 30°C - 40°C) on the Research Lab Table in the Faculty of Pharmacy and Foods (Havana University), ground into powdered form (1 mm) and stored in airtight containers.

The extracts were prepared with the ground material (60 g) without screen extracted in a Soxhlet apparatus with 675 mL of toluene during 16 hours. The toluene extracts were concentrated and evaporated under vacuum to 200 mL at 120 rpm, a temperature of 70°C and 500 mbar. All reagents used were of analytical grade (Merck). All solvents were degassed prior to use in an ultrasonic bath without filtration.

2.2 GC-MS Analysis

For the identification of metabolites present in the rhizomes, the sample were subjected to chromatographic analysis in equipment GC/MS, brand Shimadzu QP2010, equipped with a splitter split/split less. With a BP5 ($30 \text{ m} \times 0.25 \text{ mm} \times 0.25$ microns) capillary column under the following chromatographic conditions: Helium gas carrier obtained by electron impact

fragments to a power of 70 eV rate of 1.2 mL/min, 1:50 split flow and the volume of injected sample of 1 ul. Programmed oven temperature: initial temperature was 70°C with a heating ramp of 10°C/min to 300°C and remained stable at this temperature for 10 minutes. Subsequently the temperature was increased at a rate of 10°C/minute to 300°C for a total time of 78 minutes with an injector temperature 250°C and the interface temperature 300°C. The compounds were analyzed using GC/MS NIST21 and NIST107 library and having into account the results obtained after phytochemical screening according with González et al., 2017. Silylation agent was *N*, *O*-bis (trimethylsilyl) trifluoroacetamide (**BSTFA**) CAS 25561-30-2 Lot: 0901-1 Macherey-Nagel GmbH & C. KG (Figure 2).



FIGURE 2: GC-MS Shimadzu QP2010, BP5 capillary column and derivatization process of the sample used in the experiment.

2.3 Data Analysis

Comparison of the spectra with the NIST database using a probability-based matching algorithm was performed to achieve compound identification, along with comparison of relative retention indices (RI) to literature and standard reference values.

III. RESULTS AND DISCUSSION

3.1 Chemical characterization by GC-MS

Figure 3 shows the TIC chromatogram with the retention times of different kind of chemical components present in the sample, indicating that the main amount of chemical compounds in the extract are between 14.2 and 54 minutes of retention time. The most intense peaks are between 27.5 and 31.5 minutes of retention time.

After 78 minutes of running, 893 chemical components were automatically identified from the sample, and among them, the presence of 11 carboxylic acids, 13 reductants sugars and/or their derivatives, one coumarin derivative, 3 steroids and their derivatives, one catechin and one flavonoid were tentatively characterized.



FIGURE 3: TIC chromatogram of rhizomes from S. domingensis Willd.

Table 1 summarize the peaks of the main components found in the extract with their retention times (Rt) and their corresponding names. The first 23 chemical compounds were discarded because they are related with the Silylation agent and their derivatives and water, as well as, the last 20 chemical components.

ſ				Dominio	
Peak #	R. time	Molecular formula	Molecular weight	Base Peak	Library ID/Compound name [*]
1	14.220	$C_{12}H_{32}O_3Si_3$	308	73	Trimethylsilyl ether of glycerol
2	15.646	$C_{10}H_{22}O_4Si_2$	262	147	Butanedioic acid (Succinic acid)
3	16.085	$C_{12}H_{30}O_4Si_3$	322	73	Propanoic acid, 2,3-bis [(trimethylsilyl)oxy
4	19.310	$C_{13}H_{30}O_5Si_3$	350	73	Malic acid
5	20.623	$C_{14}H_{32}O_5Si_3$	364	73	3-Hydroxy glutaric acid
6	20.795	$C_{11}H_{16}O_3Si$	224	209	Benzoic acid, 4-[(trimethylsilyl)oxy]-, methyl ester
7	21.405	$C_{12}H_{28}O_3Si_2$	276	73	Hexanoic acid, 2-[(trimethylsilyl)oxy]-, trimethylsilyl ester
8	23.231	$C_{11}H_{26}O_3Si_2$	262	73	Butanoic acid, 3-methyl-2 [(trimethylsilyl)oxy]-TMS
9	27.720	$C_{20}H_{52}O_5Si_5$	512	73	Ribitol, 1,2,3,4,5-pentakis-O-(trimethylsilyl)-
10	28.601	$C_{14}H_{32}O_5Si_3$	364	73	D-Arabinonic acid, 2,3,5-tris-O-(trimethylsilyl)-, .gamma lactone
11	28.875	$C_{19}H_{46}O_6Si_4$	482	217	Glucofuranoside, methyl 2,3,5,6-tetrakis-O- (trimethylsilyl)-, .alphaD-
12	29.061	$C_{21}H_{52}O_6Si_5$	540	217	D-Fructose
13	29.205	$C_{17}H_{42}O_5Si_4$	438	217	D-Ribofuranose, 1,2,3,5-tetrakis-O-(trimethylsilyl)
14	29.486	$C_{18}H_{44}O_5Si_4$	452	73	2-Deoxy-galactose, tetrakis(trimethylsilyl)
15	29.621	$C_{21}H_{52}O_6Si_5$	540	217	betaD-Galactofuranose
16	39.241	$C_{21}H_{52}O_6Si_5$	540	73	D-Galactose
17	30.391	$C_{21}H_{52}O_6Si_5$	540	73	Glucose
18	30.575	$C_{21}H_{50}O_6Si_5$	538	73	Inosose-2, 1,3,4,5,6-pentakis-O-(trimethylsilyl)-
19	30.764	$C_{24}H_{62}O_6Si_6$	614	73	Trimethylsilyl ether of glucitol
20	31.405	$C_{21}H_{52}O_5Si_5$	524	73	Myo-inositol, 5-deoxy-1,2,3,4,6-pentakis-O- (trimethylsilyl)
21	32.470	$C_{12}H_{28}O_3Si_2$	276	73	Pentanoic acid, 4-methyl-2-[(trimethylsilyl)oxy]-, trimethylsilyl ester
22	33.185	$C_{20}H_{40}O_2$	312	88	Octadecanoic acid, ethyl ester
23	33.400	$C_{21}H_{40}O_2Si$	352	73	9,12-Octadecadienoic acid (Z,Z)-, trimethylsilyl ester
24	33.450	$C_{21}H_{42}O_2Si$	354	73	Oleic acid
25	42.570	$C_{17}H_{14}O_4$	282	282	Coumaran-3-one, 6-methoxy-2-[4-methoxybenzylidene]
26	44.290	C ₃₂ H ₅₆ OSi	484	83	Stigmasterol trimethylsilyl ether
27	45.265	C ₃₉ H ₅₄ OSi	458	129	Cholesterol trimethylsilyl ether
28	46.555	$C_{34}H_{52}O_{3}$	508	327	Cholestan-7-ol, 8,14-epoxy-3-(phenylmethoxy)-
29	47.600	$C_{30}H_{54}O_6Si_5$	650	73	Catechin, penta-TMS-ether
30	48.280	$C_{23}H_{22}O_{6}$	394	192	Rotenone

 TABLE 1

 CHEMICAL COMPONENTS CHARACTERIZED BY GC-MS FROM TOLUENE EXTRACT IN RHIZOMES OF S.

 DOMINGENSIS.

*The nearest library standards.

Preliminary phytochemical screening suggested the presence of flavonoids, alkaloids, coumarins, catechins, pirochatecolic tannins, fat and/or volatile oils, saponins, triterpens and/or steroid, quinones and reducing sugars, and the absence of resins, amino acids or amines, cardiotonic glycosides, anthocyanidins and astringents and/or bitter principles Data referred here refer to evaluations with wild material in our country (Yaque et al., 2017).

Alanine (L-Alanine or alpha-Aminopropionic acid) with Molecular formula $C_3H_7NO_2$, Molecular weight 89 and Base peak 44 was detected at 12.315 min of retention time, but the coincidence index was low (only 39 %) (Figure 4). A Glycine derivative (Glycine, N-formyl-N-(trimethylsilyl)-, trimethylsilyl ester), with MF $C_9H_{21}NO_3Si$, MW 247 and BP 73, was detected at 19.425 min of Rt, with a coincidence index of 60 % (Figure 5). Although the percentage of coincidence are different in both cases, and are not above 85-90 %, we are not discarding the presence of aminoacids in the Cuban rhizomes of *S. domingensis*.



FIGURE 5: Mass spectrum detected by GC-MS of Glycine, N-formyl-N-(trimethylsilyl)-, trimethylsilyl ester.

Contrary to expected according to the literature, sarsapogenin and smilagenin were not detected by GCMS analysis, according to the molecular masses of known saponins from *S. officinalis* (Cáceres et al., 2012), but in this case, our results confirm the presence of different steroidal compounds like stigmasterol and cholesterol, corroborating the results found by Cáceres et al., 2012 in Guatemala.

This new phytochemical profile shows the matches of 11 carboxylic acids and 6 reductants sugars or their derivatives (~34 %), and the presence of two new compounds that belongs to flavonoids group: Catechin and Rotenone, one coumarin derivative and three steroidal components or their derivatives comparing with the previous results reported by our research team in ethanolic extracts of this part of the plant (Figure 6).



Stigmasterol trimethylsilyl ether

Cholesterol trimethylsilyl ether

Cholestan-7-ol, 8,14-epoxy-3-(phenylmethoxy)

FIGURE 6: New chemical components found in S. domingensis by GC-MS in Cuba.

IV. CONCLUSIONS

GC-MS is frequently applied to characterize the chemical complexity of analytical samples based on its separation and identification capacity. Following solvent extraction and derivatization, 30 metabolites from different chemical groups can be characterized in one analytical run with a high percentage of coincidence of those compounds characterized by the same methodology in ethanolic extract from this part of the plant, principally sugars and/or derivatives and carboxylic acids. Six new phytochemical components were tentatively detected, although the authors not discarding the presence of two chemical compounds that belongs to aminoacids group. The results from plant research to exemplify the applicability of GC-MS

profiling and concurrent detection and identification of six principal groups of chemical components and other cyclic structures. Based on experimental data from own research, the present research has emphasized the capabilities of GC-MS to deduce chemical information on diverse compounds found in complex mixtures of plant metabolites. The compounds identified can be also used as biomarkers especially for *S. domingensis* due to little research has been published for this species. Further studies are needed to establish the molecules responsible for the chemical composition and the biological activities attributed to this rhizome, specially using HPLC-MS or LC-NMR. Saponins still remain indeterminable in extracts from *S. domingensis* in our country.

CONFLICT OF INTEREST

The authors declare that they have not conflict of interest.

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Experimental and Numerical Study of the Reinforced Panels Subjected to Tensile Loading: Crack Stoppers

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Abstract— The present study concerns with the experimental and numerical investigation of crack stoppers ahead of an edge crack in panels subjected to tensile loading. Two different patches (rectangular and semi-annular patches) have been analyzed. The patches (aluminium and steel) are placed at different distance from the crack, symmetrically on both sides of the panel and at a finite distance ahead of the crack tip. A finite distance ahead of the crack tip reveals that depending on the distance, the crack tip could remain straight or curve. In such cases, the crack could either be arrested, run through or run around the reinforcements. Moreover, the degree of instability is reflected by an index parameter that accounts for the effect of load, geometry and material properties. Moreover, a geometrically nonlinear, two-dimensional (2D) finite element analysis (Comsol Multiphysics) has been employed to determine the local energy intensity. It would be of special interest to know whether the crack would run straight and arrest at the patch regardless of the other variables. The ultimate goal for straight crack path is to produce sufficient low local energy intensity. This gives a significant advantage because as the local energy intensity is increased, crack would tend to curve and lead to complete fracture of the patched specimens. It is equivalent of moving the patch closer to the crack tip. The predictions made from the strain energy density theory, as well as, there is a good agreement between finite element results and experimental findings.

Keywords— Comsol multiphysics, crack stoppers, energy density, reinforcement patch.

I. INTRODUCTION

A challenging problem which arises during production are the defects in structural components. A practice often used is to repair the cracked members by bonding patches to redirect the load path [1-8] to reduce the local stress or energy intensity level below critical. However, it is well known, that unlike the repair patches [9–15], which are used when fatigue cracks are detected, crack retarders will be part of the original aircraft structure and subjected to operational loads and environments throughout the entire service life. It means that aircraft structures could be safer, lighter, and cheaper. Especially, by using adhesively bonded composite patches, which are more efficient and much less damaging to the parent structure than standard repairs based on mechanically fastened metallic patches [16].

The development of bonded composite repair technology has been accelerated by many researches in the aerospace industry in the interests of increasing the service life and reducing the repair cost [17–19]. Composite repair has taken numerous roles on military aircraft, such as reinforcement in areas where stress corrosion cracking can occur, as on the lower surface of the wing pivot on a U.S. Air Force F-111, or fatigue cracking around a fuel decant hole on a Royal Australian Air Force (RAAF) Mirage III. On commercial aircraft, the use of composite repairs is in early stages [20]. On the other hand, one of the key issues in aircraft failure is the presence of multiple cracks in a local area, known as multisite damage (MSD). Analysis of the repair of MSD has been proposed by Park et al., [21] by placing a patch over a cracked pin-loaded fastener hole.

In addition to knowledge of the detailed stresses near the defects or cracks, a suitable failure criterion is needed to determine the effectiveness of the repair process. Improper repair could cause more harm than good. This has been known in the aircraft repair industry. The classical failure criteria are not adequate because they could not correct local and global failure in a constant manner to include the combined effects of loading rate, geometry and material. The concept of stable failure is pertinent, which means that structures in order to be safe, must be designed in a way that cracks are developing smoothly and slowly according to the increase in loading rate. The damage should become so obvious that repair be made prior to catastrophic failure. More specifically, the period of stable crack growth should be prolonged. To this end, the local and global stationary values of the strain energy density function can be used mostly effectively for this purpose. While the basic concept of this approach can be found in [22-24], applications to a host of practical problem are given in [25-31]. The site of crack initiation can be determined without the need to assume an initial crack; it is taken to coincide with regions where dilatation is largest in comparison with distortion. These regions can be sorted out automatically from the maximum and minimum value of the strain energy density function. Hence, the approach applies equally well to nonlinear materials [31] where the dilatational and distortional components of the strain energy density function can no longer be separated as in the case of linear elasticity. To illustrate the application of these criteria, it suffices to consider linear elasticity.

Another interesting parameter is the stress intensity factor. The determination of the stress intensity factor (SIF) at the crack tip for any loading mode (K_I , K_{II} , K_{III}) is one of the means for analyzing the performance of the bonded patch in repairing cracks [32–39]. On the other hand, several authors computed the SIF at the crack tip of repaired cracks under mechanical loading [40-50]. Nowadays, stress intensity factor can be calculated with great accuracy by using finite element software's. A quite interesting example is Bachir Bouiadjra et al. [51] report, where for a single-patch repair analysis the increase of the patch thickness by about 50% reduces the stress intensity factors at the same order. They suggested that in order to increase accuracy multiple layers of bonded composite patch, must be used.

Among the means that can strengthen these ideas is the use of the double-sided symmetric patch. Many authors showed the advantage of the double-sided symmetric patches experimentally and numerically, among them, one can quote: Ting et al. [52], Bachir Bouiadjra et al. [32, 43, 53] and Belhouari et al. [47]. The literature review [32, 43, 47, 52, 53] shows that the double-sided symmetric patch reduces more the stress intensity at the crack tip, while it annuls the bending effect due to the eccentricity of the patch. The single edge crack configuration is depicted in this work where patches of different shape, size, location and material are bonded to the specimens. Evaluated are the corresponding strain energy density functions from which the location of the local and global stationary value can be determined. This yield a single parameter that would reflect the combine influence of the load, geometry and material. The magnitude of this parameter determines the overall stability or instability of the patched structure. Experiments are then performed for steel and aluminum patches of different size and patch location to study whether the analytical findings would yield the same results.

II. REINFORCEMENTS AND MATERIALS

2.1 Geometry Configuration

The testing configuration is a rectangular panel with an edge crack of length a as shown in Fig. 1. Following, Zacharopoulos report [54], a rectangular panel with a certain dimension was selected. The dimensions of the specimen were as following; width W=6cm; a height 2H=20cm and thickness $t_P=0.24$ cm. The panel is subjected to a uniform stress ($\sigma = 10$ MPa) which is applied in the y-direction, that is normal to the crack which coincides with the x-axis. A central crack of length 2a perpendicular to the loading axis exists in the plate. The rectangular and annular patch have dimensions of r = 1 cm, b = 1 cm and $t_A = t_S = 0.1$ cm where **A** stands for aluminum and **S** for steel. The parameter d in Fig. 1 is used to denote the distance from the patch to the crack tip.



FIGURE 1: A test specimen configuration: (a) plane view; (b) thickness view

2.2 Material and Fracture Properties

Two different patches materials (aluminium and steel) were considered in this work, while the panel is made of plexiglass. The mechanical and fracture properties of the materials are given in Table 1. The effect of the patches and panel can be regarded to behave as a unit with an effective stiffness.

TABLE 1		
MECHANICAL AND FRACTURE PROPERTIES OF PANELS AND PATCHES [54]	

Material	Young's Modulus E (MPa)	Poisson's Ratio, v	Yield Strengtho _y (MPa)	$\begin{array}{c} Tensile \ Strength \\ \sigma_{f} \ (MPa) \end{array}$	$ Critical Stress Int. Factor \\ K_{IC}(MPa \ m^{1/2}) $
Plexiglass	3400	0.31		85.0	1.19
Aluminum	71000	0.33	72.0	150.0	
Steel	210000	0.28	200.0	370.0	

Let E* denote the effective Young's modulus given by (1)

$$E^* = \frac{t_P E_p + 2t_j E_j}{t_P + 2t_j}$$
(1)

where E_P and E_j are, respectively, the Young's moduli of plexiglass and patches. The same applies to the estimate of an effective Poisson's ratio (2):

$$v^* = \frac{t_P v_p + 2t_j v_j}{t_P + 2t_j} \tag{2}$$

In which v_p and v_j stand for Poisson's ratio of the panel and patches, respectively. Based on Eqs. (1) and (2), the effective values E^{*} and v^{*} are calculated and given in Table 2.

 TABLE 2

 EFFECTIVE PROPERTIES OF E* AND v*[54]

Material	E (MPa)	v
Plexiglass/ Aluminum	34000	0.309
Plexiglass/Steel	97000	0.291

Furthermore, for plexiglass, the value $r_c = 2.8 \times 10^{-3}$ cm is used to compute for K_{IC} in Table 1 from Eq. (2).

2.3 Failure Path

It is well known that for a body with an initial crack, the site of maximum strain energy density function would coincide with the crack tip. That means, there is a location where the volume changes, making the strain energy per unit volume be smallest one dW/dV. In order to intedify the location, consider a typical location P_j with a local coordinate (x_j, y_j) (fig. 2). The distance r_o varies as a function of the angle θ_j . As the distance increases the subscript L would be different as the position of the reference axis changes. That means, for every value, another value of $(dW/dV)_{min}^{max}$ would be found when reference is made to the global coordinates (X, Y) in Fig. 2.

Moreover, as the L tends to G, the phenomenon would be called as a global, and it could be tested also experimentally. Hypothesis of the Maximum Gradient of the Strain Energy Density (HMG of SED), which predicts with great accuracy the directional accuracy has been applied to brittle materials or materials that behave in brittle way (elastic-plastic materials) [55]. There is also a considerable amount of literature on strain energy density theory [22-24], but this study, will be limited to fracture only, by using also finite element analysis (Comsol Multiphysics) in order to verify the experimental results. As for the instability index, namely 1 [2,22-26], it could be taken approximately as a straight-line distance between L and G. This would provide an indication of the fracture trajectory at which stable growth would terminate at G where the onset of rapid fracture would start. For a more precise estimate of the crack path, small increments of crack growth can be taken to obtain the full curvature of the path from L to G as shown in Fig. 1. Such analysis for crack trajectories emanating from the tip of an angle crack can be found in [56].



FIGURE 2: System ability: local and global stationary values of strain energy density.

III. EXPERIMENTAL PROCEDURE

A fretsaw is first used to make a cut in the plexiglass and a finer cut is then made using a razor blade. The specimen is loaded under control-displacement condition and the edge notch is allowed to propagate stably until the desired length is reached. Both aluminum and steel are used to make the rectangular and circular patches with $t_A = t_S = 0.1$ cm. The planar dimensions for the former are b = 1 cm and h = 2 and 4 cm while for the latter r = b = 1 cm. These patches are bonded to both sides of the panel using a thin glue film with shear strength of 21.0MPa. Such strength is sufficient to transfer the load from the plexiglass to the metallic patches without debonding.

Moreover, the specimens were tested under load-controlled condition in a 60 kN Shenck hydraulic machine. Loading rates are increased to observe whether the crack propagation characteristics would alter for different patch length. All results of critical stresses for patched panels are normalized with reference to the fracture stress of the unreinforced panel with an edge crack. Hence, the ratio would always be greater than unity. While discrete test data points are obtained, all analytical results are given by solid curve.

IV. FINITE ELEMENT ANALYSIS

To ensure there is no relative motion between the plate and the patch, both of them are constrained by tying with the adhesive layer. The finite element analysis was performed using Comsol Multiphysics [57]. It has been applied to solve for the stresses and strain energy density functions. Numerical results are obtained for the rectangular and semicircular patches, the dimensions of which have already been given. For the aluminum patch $E_A/E_P = 21$ while for the steel patch $E_S/E_P = 62$. The distance d = 1, 2 and 3 cm for a = 1 and 2 cm (rectangular patches).



FIGURE 3: Finite element mesh density for semi-circle and rectangular reinforcement specimen [145,535 elements for all cases]

4.1 Rectangular Patches

Plotted in Figs. 4-7 are the normalized critical stresses σ_j/σ_c (j = A, S) as a function of the distance d. All curves tend to unity at about d = 3cm at which point the crack has no interaction with the patch. The critical stress σ_j and σ_c correspond to the panel with and without the patch, respectively. First of all, the test data agree well with the solid curves obtained from the strain energy density theory. The results in Figs. 3 and 4 correspond to those for the aluminum and steel patch, respectively. Note that when the patch size is doubled, the critical stress ratio σ_j/σ_p for aluminum and steel patch increases 35% and 45%, respectively, for patch location of d = 1 cm (a=1cm). The effect of reinforcement is significant and is expected to decrease with increasing d. The increase of critical stress due to enlargement of the patch size are clearly shown in Figs. 4 and 5 for the case of a = 1 cm. Similar results are displayed in Figs. 6 and 7 for a = 2cm.



FIGURE 4: Normalized critical stress versus distance d for aluminum rectangular patch and a=1cm. (Note: a in the text is visualized as ``a'' in the figures throughout.)



FIGURE 6: Normalized critical stress versus distance d for aluminium rectangular patch and a=2cm.



FIGURE 5: Normalized critical stress versus distance d for steel rectangular patch and a=1cm.



FIGURE 7: Normalized critical stress versus distance d for steel rectangular patch and a=2cm.

Size and location of the patches also have a significant influence on the critical stresses. As the patch size is doubled, the critical stresses for panels differs for each patch. For instance, with aluminum and steel patches, the critical stresses would increase 50% and 15%, respectively, at a=1cm and d = 1 cm. Plotted in Fig. 8 are the stress ratio σ_j/σ_c versus the thickness ratio of the patch t_j/t_p . Except for the initial rise of gain in stiffness, the effect tapers off quickly. This means that any further increase in patch thickness would not increase the critical stress.



FIGURE 8: Normalized critical stress versus normalized thickness of rectangular patch for two different materials and a=1cm.

4.2 Rectangular Patches: Crack Trajectories and Instability

Since the geometry and material are symmetric across the crack plane, crack is expected to propagate straight ahead. The straight path of the crack tip which appears in macroscale, is practically insensitive in any small fluctuations and possible multi-branching, which are took place in microscale and can owe in the irregularities of microstructure. Even small imperfections by the manufacture of initial crack tip, do not influence the rectilinear trajectory propagation of crack tip [54].

Fig. 9 plots the strain energy density function as a function of the distance r ahead of the crack for d = 1 and 2 cm, where dW/dV would decrease with increasing distance from the crack. Displayed in Figs. 10 and 11 are results for the instability index as a function of the distance for a = 1 and 2 cm, respectively. In both cases, 1 tends to increase monotonically. This means that crack instability would increase with the distance d. The same is found for both the aluminum and steel patch. Fig. 12a, b shows the constant contours of dW/dV for the edge crack panel without any patches with a = 1 cm. The point G lies inside of the specimen (finite element result). This implies that stable crack propagation prevails regardless of loading rate as verified by experiments. If a patch is bonded to the specimen with the inner edge at d = 3 cm while the other edge is 1 cm from the outer edge of specimen. In this case, G is found to be at a distance 2.9cm from the crack tip (Fig. 12b). If the load increases further, the crack trajectory will run around the perimeter of the reinforcement patch. Similar, results will be displayed by moving the same patch closer to the crack tip, at d = 2 cm. In this case, it would tend to increase the intensity of the local energy intensity and moves G away from a straight path (Fig. 13).







FIGURE 10: Variation of instability index l with distance d for rectangular patch with a=1cm (aluminium).



FIGURE 11: Variation of instability index l with distance d for rectangular patch with a=2cm (aluminium).



FIGURE 12: (a) Constant contours of strain energy for edge-cracked panel (a) without patch and a=1cm (b) with steel rectangular (bxh=1x4) patch with d=3cm and a=1cm-Damage representation.

Furthermore, the damage of the specimen tends to move away from the patch (R) before the lower corner (fig. 13). This was observed by tests where the crack would curve followed by complete fracture of the specimen. In that case, the curved trajectory will be unstable and perhaps not unique. It is clear that until the critical point (fig. 13) the crack propagates stably but when begins the divergence of the tip from its original axis, then is moved more rapidly, and also with unstable way. Figs. 14a and b exhibit the location of G for patches with size 1 x 2 cm² and 1 x 4 cm², respectively, at the same location d = 1 cm.

Note that G is shown to be at the boundary of the patch for both figures 14a and b where the crack, again, tends to run around the edge of the patch. These predictions from the strain energy density theory were observed also during experimental procedures. The curved trajectory which arises, generally is unstable but for small values ratio (H/W), this tend to become stable. The foregoing results for the aluminum and steel patches are summarized in Tables 3 and 4, respectively.



FIGURE 13: Damage for edge-cracked panel with steel rectangular patch (bxh=1x4, a=1cm) with d=2cm and d=3cm, respectively.



FIGURE 14: (a) Constant contours of strain energy for edge-cracked panel (a) with aluminium rectangular patch (bxh = 1x2) with d=1cm and a=1cm(b) with aluminium rectangular patch (bxh=1x4) patch with d=1cm and a=1cm-Damage representation.

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d (cm)	$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 2 \mathbf{cm}^2$		$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 4 \mathbf{cm}^2$		
a=1cm	l (cm)	Crack Path	l (cm)	Crack Path	
1	1.95	Curve	1.90	Curve	
2	2.75	Curve	2.95	Curve	
3	3.90	Straight	3.93	Curve	
a=2cm					
1	2.95	Curve	2.85	Curve	
2	3.98	Straight	3.85	Straight	
3	4.80	Straight	4.80	Straight	

TABLE 3 INSTABILITY INDEX FOR STEEL PATCH WITH $E_s/E_p=21$

TABLE 4
INSTABILITY INDEX FOR STEEL PATCH WITH $E_s/E_P=62$

d (cm)	$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 2 \mathbf{cm}^2$		$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 4 \mathbf{cm}^2$	
a=1cm	l (cm)	Crack Path	l (cm)	Crack Path
1	1.85	Curve	1.60	Curve
2	2.82	Curve	2.92	Curve
3	3.83	Straight	3.90	Curve
a=2cm				
1	2.90	Curve	2.80	Curve
2	3.80	Curve	3.95	Curve
3	4.80	Straight	4.80	Straight

4.3 Semi-Annular Patches: Critical Stresses

Shown in Figs. 15 and 16 are decrease of the normalized critical stresses with the distance d for initial crack length a = 1 and 2 cm, respectively. It can be seen from these results that considerable gain in the critical stress level can be realized for the steel patch by having it near to the crack. At about d = 3 cm, the effect of the path tends to diminish. The influence of the patch thickness on the critical stress is illustrated in Fig. 17. The initial gain tapers off with continuous increase in t_A and t_S , a result that is similar to the rectangular patch and is to be expected.





FIGURE 15: Comparison of predicted normalized critical stress versus distance d for semi annular patch for a=1cm.





FIGURE 17: Normalized critical stress versus normalized thickness of semi annular patch for two different materials and a=1cm.

4.4 Semi-Annular Patches: Crack Trajectory and Instability Index

For a = 1 cm, Fig. 18 shows that rises with d and is independent of the material of the patches. A small difference for the aluminum and steel path is observed as the initial crack length is increased to 2 cm. This is shown in Fig. 19. Contours of constant dW/dV are displayed in Fig. 20 for a = 1 cm and d = 2 cm. In this case, G remains on the axis of symmetry with l = 1.80 cm. Similar results are found for d = 1 and 3 cm and a = 2 cm and hence no further discussions are required. Refer to Table 5 for different value of the instability index. Similar results are found for d = 1 and a mathematical constant for d = 1 and a mathematical constant for d = 1 and a mathematical constant a = 2 cm and hence no further discussions are required. Refer to Table 5 for different value of the instability index.





FIGURE 18: Variation of instability index l with distance d for semi annular patch and a=1cm

FIGURE 19: Variation of instability index l with distance d for semi annular patch and different materials with a=2cm.

INSTABILITY INDEX FOR ALUMINIUM AND STEEL PATCH					
d (cm)	$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 2 \mathbf{cm}^2$		$\mathbf{b} \mathbf{x} \mathbf{h} = 1 \mathbf{x} 4 \mathbf{cm}^2$		
a=1cm	l (cm)	Crack Path	l (cm)	Crack Path	
1	0.90	Straight	0.62	Straight	
2	1.80	Straight	1.90	Straight	
3	2.80	Straight	2.80	Straight	
a=2cm					
1	0.90	Straight	0.90	Straight	
2	1.92	Straight	1.90	Straight	
3	2.90	Straight	2.92	Straight	

 TABLE 5

 Instability index for aluminium and steel patch



FIGURE 20: Constant contours of strain energy density for edge cracked panel with steel semi annular patch with d=2cm and a=1cm.

V. CONCLUSIONS

This work has conclusively proven that the instability index can be used as an indicator of the effectiveness of reinforcement to arrest a crack. Several parameters, such as geometric and materials of the patch alters effectively is the intensity of the load transferred to the crack tip region and hence the crack growth characteristics. Broadly translated our findings indicate that the edge crack specimen gives rise to unstable fracture under uniform tensile loading. However, the kind of the crack propagation, depends on the rate and the way of loading of the specimen from the test machine, namely, whether the increase of the imposed displacement or the force respectively, will be controlled.

The evidence from this study that, for low local energy intensity of the rectangular patch specimens, the initial crack runs straight and arrests at the patch edge regardless of the initial crack length and position. However, as the patch is moved closer to the crack tip crack path deviated from the straight line (critical point). This occurred for the rectangular patches regardless of the other variables. A temporary arrest of extension is presented at point G, but with furthermore increasing of the loading, leaded to catholic fracture. Moreover, the crack trajectory was straight for the semi-annular patch even when patch distance from the crack tip is increased, a new crack trajectory was observed. This is independent of the geometrical characteristics of the specimen, as well as, the initial crack length. Nevertheless, the crack tip is still trapped inside the semi-annular patch for a certain load. Our work has led us to conclude that, we can direct to a certain point outside the patch zone. That means, a further investigation of the method of Hypothesis of the Maximum Gradient of the Strain Energy Density (HMG of SED)

must be investigated. The most important limitation lies in the fact that this method provides great accuracy only in a smallscale specimen. It is also recommended that further research should be undertaken in the area of ductile materials.

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