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THE USE OF LIGNIN FOR ENVIRONMENTAL PROTECTION: AN OVERVIEW OF RECENT LITERATURE

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ABSTRACT

Reducing environmental pollution is a particularly important issue, intensively studied nationally and internationally, as evidenced by the large number of scientific papers published on this topic. Literature studies show that reducing environmental pollution means not only reducing the amount of air / water / soil pollutants but also finding ways to recover waste from industrial activities that will reduce the problems of environmental pollution due to their storage.

The materials resulting from the gasification processes of cellulose waste, as well as the materials obtained with the help of cellulose extracted from cellulose waste, were tested in the adsorption processes of some polluting species and the results demonstrated the efficiency of these materials for reducing the environmental pollution. The use of lignin as an adsorbent for the removal of pollutants (organic dyes or metal ions) from industrial wastewater can be considered a viable alternative, which can be a solution to both the problem of lignin recovery and the reduction of pollutant content in industrial wastewater.

KEYWORDS: lignin, heavy metals, dyes, cellulose waste, pollution

1. Introduction

In addition to environmental pollution caused by the development of anthropogenic activities, referred to in the literature as permanent pollution, in recent years, both worldwide and at European level, there has been an increase in the number of pollution phenomena caused by technological accidents, from various branches of industry, car and rail transport or the mismanagement and/or irrational management of liquid and solid waste from domestic activity [1, 2].

Legislation in many parts of the world severely penalizes environmental pollution. The mining industry, the metallurgical industry, and the textile industry (especially textile chemical finishing) have felt drastically the rigors of the new requirements of the legislation regarding environmental protection [1-3].

Due to their relatively high solubility, a wide variety of pollutants (metal ions, phenols, or organic dyes) easily reach industrial wastewater and their content in such effluents depends on the type of industrial activity and the nature of the technological process applied [5-9].

Numerous methods are presented in the literature for the removal of metal ions and dyes from industrial wastewater: physical processes (membrane filtration, flotation); simple chemical processes (chemical precipitation, coagulation, ion exchange, electrolysis, solvent extraction, adsorption); use of biologically active microorganisms [1, 2-9].

Studies in the literature on the reduction of environmental pollution have shown that a number of materials, which have a porous structure and a relatively large number of superficial functional groups, can be used effectively to remove pollutants from aqueous solutions. Thus, natural materials or various categories of agricultural or industrial waste, generically called low-cost adsorbents can be successfully used for the removal of heavy metal ions and organic substances from wastewater [3-5, 10-12]. Adsorption is an efficient method for removing dyes, heavy metals, and phenol derivatives [10-13].

The lignocellulosic materials can be used as efficient sorbents for the removal of different pollutants from wastewaters (heavy metal ions, dyes, and other organic compounds), due to their fundamental characteristics, such as high porosity and specific surface area, good mechanical resistance,

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tolerance to biological adsorbed solid layers [1, 2, 4-12].

Lignin constitutes one-third of biomass and this component is typically burned to produce heat and electricity within paper mills and biorefineries [3, 13]. For many years, lignin was considered just a waste material or a very low-grade by-product of the pulping process. The use of lignin as an adsorbent for the removal of pollutants (organic dyes or metal ions) from industrial wastewater can be considered a viable alternative, which can be a solution to both the problem of lignin recovery and the reduction of pollutant content in industrial wastewater. Lignin is a natural polymer, which is abundantly present in cell walls of terrestrial plants and acts as a binding agent [2, 13-16]. Infrared spectroscopy is the most suitable analysis method for identifying the presence of polar functional groups in the molecule structure of organic compounds. Lignin contains many functional groups: hydroxyl, carbonyl, methoxyl, carboxyl, and the Infrared spectrum clearly indicates their presence in the molecule (Table 1); the FTIR spectra of lignin include the most extensive set of stretching and bending vibration bands of them. These functional groups can interact with ionic species (organic or inorganic) present in wastewater, making this natural material useful in adsorption processes [1, 2, 14]. The removal of different pollutant species by adsorbents based on natural or modified lignin and/or lignocellulosic materials has been intensively studied (Tables 2, 3) [1, 2, 13-20]. The results suggested that the adsorption of inorganic and organic pollutants on lignin is a progression towards a perspective method.

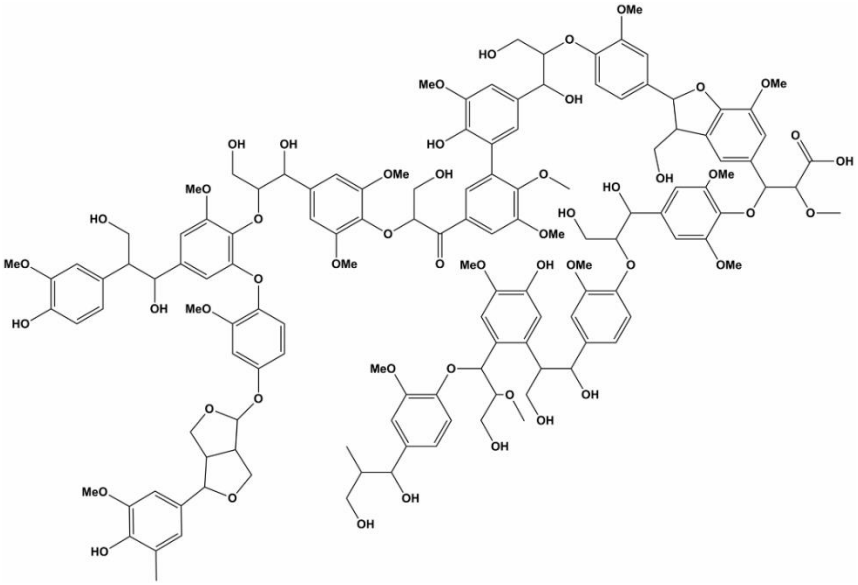

Fig. 1. *Lignin structure* [1, 3]

Table 1. *FTIR spectrum of lignin* [1-3, 13]

IR Absorption band (cm^{-1})	Type of vibration
3500-3400	Stretching vibration of alcoholic and phenolic –OH groups
2920-2840	Stretching vibration of C–H bonds in $–CH_3$ and $–CH_2$ groups
1715-1710	Stretching vibration of C=O bonds (in –COOH group)
1515-1500	Aromatic ring vibrations
1130-1035	Deformation vibrations of aromatic C–H bonds and C–O stretching for secondary alcohols

2. The use of lignin as an adsorbent for the removal of pollutants

From a chemical point of view, lignin is considered an amorphous crosslinked polymer, which has a three-dimensional aromatic structure and it contains many functional groups, such as hydroxyl, carbonyl, methoxyl, and carboxyl. These functional groups can interact with ionic species present in aqueous media, making this material useful in adsorption processes [1, 2, 13, 14].

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The presence of these functional groups and the porous structure of lignin, demonstrated by microscopic studies, show that this material can be used as an adsorbent to retain pollutant species from aqueous solutions and can therefore be used in processes to reduce environmental pollution [2, 12-14].

There are numerous data from the literature attesting to the quantitative retention of organic dyes on lignin. Due to this behavior, lignin differs significantly from cellulose, on which dyes cannot be permanently fixed [1, 15].

Regardless of the nature of the dye used, their adsorption on lignin is a relatively complex process, the efficiency of which depends on a number of experimental parameters, such as: the pH of the solution, adsorbent dose, initial concentration of the dye in the solution, contact time and temperature [14, 16-21].

The reviewed literature demonstrated that non-hazardous materials can be used as inorganic and organic pollutants removers from industrial effluents to overcome water pollution [12, 14-21].

3. Lignin as an adsorbent for the removal of pollutants. Adsorption studies and mathematical modelling

Adsorption equilibrium data are important to provide physicochemical information to explain the mechanism of adsorption. The absorption capacity of an adsorbent can also be described by the equilibrium sorption isotherm, which is characterized by specific constants, whose values represent the surface properties and affinity of an adsorbent [12, 13-16].

Different mathematical models, generically called models of adsorption isotherms are used to interpret experimentally obtained adsorption isotherms. Langmuir and Freundlich models are most commonly used to establish equilibrium conditions in the case of adsorption processes and are considered classical models of adsorption isotherms [14-20]. The adsorbent material used in recent studies was residual lignin, obtained as waste from the pulp and paper industry, which from a macroscopic point of view is a dark brown powder [1, 2]. The wasted lignin is a huge amount, but less than 10% of this waste material was utilized and this fact is imposing as an important environmental problem [2].

There are numerous data from the literature attesting to the quantitative retention of organic dyes and heavy metals on lignin. Due to this behavior, lignin differs significantly from cellulose, on which dyes cannot be permanently fixed, regardless of whether they are acidic or basic [1, 2-5]. However, regardless of the nature of the dye used, their adsorption on lignin is a relatively complex process, the efficiency of which depends on a number of experimental parameters, such as: the pH of the aqueous solution, adsorbent dose, initial concentration of the dye in the solution, time contact, and temperature [1, 2].

Table 1. *Equilibrium adsorption data for some pollutants using lignin as adsorbent* [1, 2]

Pollutant	Langmuir Model	Freundlich Model
Brilliant Red	R^2 = 0.9980 q_m = 12.05 mg/g K_L = 0.051 L/mg	R^2 = 0.9630 K_F = 1.77 mg/g n = 2.44
Pb(II)	R^2 = 0.9979 q_m = 32.36 mg/g K_L = 0.111 L/mg	R^2 = 0.9232 K_F = 7.26 mg/g n = 2.61
Cu(II)	R^2 = 0.9979 q_m = 32.36 mg/g K_L = 0.159 L/mg	R^2 = 0.9374 K_F = 7.55 mg/g n = 2.56

In order to increase the adsorption performance of lignin in the processes of reducing environmental pollution, it is necessary to activate it and the activation treatments used in this case must be simple, so that the preparation costs of the adsorbent material remain low. One of the possibilities of lignin capitalization described in the literature is its use as fuel in order to obtain energy. Thus, the combustion of lignin can be considered an activation process because the material obtained can be used as an adsorbent for retaining polluting species from aqueous media. In addition, as a result of this activation process, significant amounts of heat are obtained, which in turn can be used for various purposes [1, 2, 20-22]. From the comparison of the experimental data presented in the literature, it can be said that thermally activated lignin has a much higher adsorption capacity for heavy metal ions and dyes in aqueous solutions compared to unburned lignin [1-3].

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Table 2. *Adsorption capacities of different natural adsorbents for heavy metal ions and dyes* [14-21]

Adsorbent type	Pollutant	Uptake capacity (mg/g)
Activated carbon	Cr(VI)	39.54 mg/g
Lignin	Cr(VI)	17.96 mg/g
Modified lignin	Zn(II)	95.0 mg/g
Chitin/lignin adsorbent	Ni(II)	88.0 mg/g
Kraft lignin	Cd(II)	137.14 mg/g
Activated carbon from pomelo peels	Malachite green	178.43 mg/g
Yellow clay	Congo Red	22.0 mg/g
Activated carbon	Reactive Blue 171	71.94 mg/g
Peat	Rodamine B	16.72 mg/g
Wood sawdust (walnut)	Methylene Blue	59.17 mg/g
Cereal chaff	Methylene Blue	20.3 mg/g

4. Conclusions

The materials resulting from the gasification processes of cellulose waste, as well as the materials obtained with the help of cellulose extracted from cellulose waste, were tested in the adsorption processes of some polluting species and the results demonstrated the efficiency of these materials for reducing the environmental pollution.

The lignocellulosic materials can be used as efficient sorbents for the removal of different pollutants from wastewaters (heavy metal ions, dyes, and other organic compounds), due to their fundamental characteristics, such as high porosity and specific surface area, good mechanical resistance, tolerance to biological adsorbed solid layers.

The use of lignin as an adsorbent for the removal of pollutants (organic dyes or metal ions) from industrial wastewater can be considered a viable alternative, which can be a solution to both the problem of lignin recovery and the reduction of pollutant content in industrial wastewater.

Lignin offers some important advantages over synthetic materials such as: biodegradability, availability in various industrial waste, it is CO_2 neutral, environmentally friendly nature, and cost-effective.

The reviewed literature demonstrated that non-hazardous materials can be used as inorganic and organic pollutants removers from industrial effluents to overcome water pollution.

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CONSIDERATIONS ON THE CURRENT STATE OF SENSORS ATTACHED TO DRONES APPLICATIONS IN ENVIRONMENTAL QUALITY MONITORING

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ABSTRACT

Environmental pollution can be detected by installing special devices on the drone, called sensors, which will collect data on air, water, and soil quality. The topic of using drones with sensors is currently known and is applied in various fields such as agriculture, meteorology, hydrology, marine and oceanographic prospecting, gas leak inspection, environmental monitoring, etc. Air, water, and soil sensors and collectors attached to drones are used to detect and collect contaminants. The remote sensing method uses multispectral optical and thermal sensors for modelling and mapping applications.

KEYWORDS: sensors, UAV, environment, pollution, monitoring

1. Introduction

Today, drones are playing an increasingly important role in the civilian field. Civilian drones are able to fly for tens of minutes and are maneuvered from the ground and from a distance. Civilian drones are controlled wirelessly via remote control or applications installed on the smartphone with an operating system. There are several types of drones that vary depending on the achieved performance and the technical characteristics: camera capabilities, flight distance, or battery life [1].

UAVs refer to a wide range of different platforms that due to their physical size and power differ in the simplicity of operation, flight endurance, and capabilities.

There are several UAVs used in various applications namely multi-rotor drones, rotary wing, and fixed-wing drones. They have the same qualities, but the multi-rotor UAV is more stable and easier to control [1].

Fig. 1. *Fixed-wing UAV, equipped with four vertical rotors, VTOL PD-1, Ukrspecsystems* [2]

Fig. 2. *Multi-rotor drone - Octocopter Predator FAE 1115* [3]

The main areas of civilian applications that use drones are [1]:

- monitoring in agriculture;
- monitoring in forestry;
- environmental monitoring
- gas leak detection;
- fire detection;
- monitoring of photovoltaic systems;
- wind turbine inspection;
- data acquisition in hard-to-reach areas;
- monitoring of constructions and buildings;
- power line control;
- aerial surveillance and photography;
- archeology.

In environmental monitoring, there are two types of sensors in a drone: flight control system sensors and sensors for collecting and analysing samples.

The selection of appropriate sensors for measuring the environmental parameters is a very

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important task. The sensors should have a short enough response time appropriate for in-flight measurements, the data exchange interface should not exceed the limits and specifications set by the airborne hardware and the price should be within reasonable limits [4].

Mobile real-time commercial micro-sensors attached to UAVs are used for the measurement of pollutants with high resolution but also capturing the dynamics of pollutants in the environment. A UAV equipped with multi-pollutant sensors requires a design that allows inserting and incorporating several sensors with distinct operating modes. Each sensor is sensitive to a particular pollutant and produces specific signals [5].

UAV remote sensing functions that should be covered by the sensors installed onboard include electromagnetic spectrum sensors, biological sensors, and chemical sensors. The UAV-mounted sensors can be utilised for air temperature and parameters, as also atmospheric conditions [6].

In environmental monitoring, current research and use aim data acquisition of air, water, and soil samples with onboard sensors. Sensors can acquire data on environmental parameters such as temperature, humidity, air pressure, pH, nitrate, ammonia, oxides and other air, soil, and water pollutants. The success of the data collection depends on the weather conditions and operating restrictions caused by the relief or urban infrastructure, but also on the performance of the sensors used in the data acquisition (image, sound, temperature, etc.) [7].

2. Applications of drones with sensors for environmental monitoring

The research revealed new applications for drones, such as collecting samples for air, soil, and water quality for laboratory analysis or assessing in situ with onboard sensors.

A drone is different from a satellite, only in that it operates at a much lower altitude. This allows for much greater detail with less atmospheric interference [8].

The near-range monitoring from drones (UAV or UAS) is a remote sensing method of the airborne oil spill. Near-range monitoring of oil spills includes mapping of relative and absolute oil layer thickness, as well as classification of the type of oil. Different types of oil sensors are: aerial photography and video, multispectral optical and thermal sensors (visible, UV, thermal-IR, UV-IR sensors), microwave radiometers, hyperspectral sensors, laser, radar and integrated airborne sensing systems. Drones can also be used to monitor localized oil pollution such as oil leakage from shipwrecks [9].

In Figures 3 and 4, the thickest emulsions show the highest heat emission (white areas in Fig. 3) while thinner emulsions appear cooler (darker) than the surrounding water due to petroleum's lower emissivity properties [10].

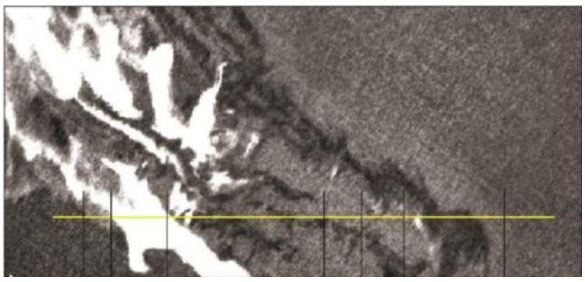

Fig. 3. *Thermal IR of a large area of emulsified oil during the Deep Water Horizon spill* [10]

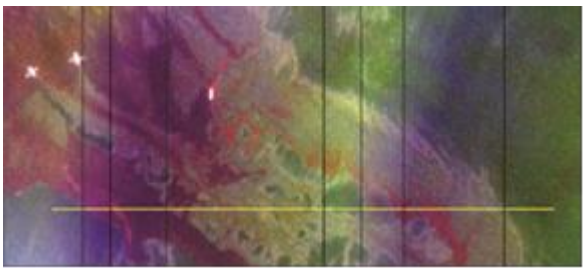

Fig. 4. *Visible multispectral sensor using 450, 551 and 600 nm bands for the blue, green and red image components* [10]

The sensor technology measures a variety of air pollutants and natural components. These have been developed for the Internet of Things (IoT) projects in which mass-produced sensors are placed throughout a city to produce real-time air quality information but are also suitable for drone platforms. The available sensors include temperature, humidity, air pressure, CO, CO_2, O_2, O_3, NO, NO_2, SO_2, NH_3, CH_4 (and combustible gases), H_2, H_2S, HCl, HCN, PH_3, Cl_2, and particulate matter (PM1, 2.5 and 10). Potential applications include: sampling pollution point sources, such as an industrial or a construction site, where air samples are taken by the drone from above a site, compared to those taken near-ground; sampling vertically can predict changes in ground-level pollution, especially of particulate matter [8].

The unmanned aerial vehicle (UAV) has a high capacity for collecting air quality data by spatial and temporal measurements with high resolution.

The vertical atmospheric measurements are very useful for evaluating and forecasting air pollution, especially in high-building metropolitan areas.

The use of unmanned aerial vehicles (UAV) has shown that its applications in air pollution monitoring studies are effective [5].

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Research in atmospheric aerosols showed the vertical profiles of black carbon concentration determined with the micro-aethalometer AE-51 mounted on a drone. Thus, the black carbon section (Fig. 5) was almost stable, with variations in some regions around 2.5 μg/m^3. The results showed that the use of drones for research into atmospheric aerosols is performing [11].

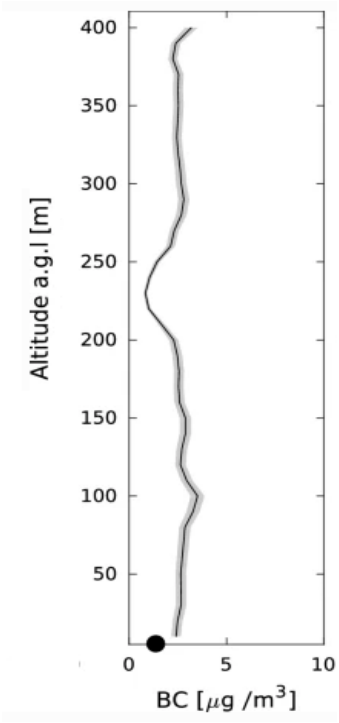

Fig. 5. *The black carbon concentration* [11]

A study achieved through a $PM_{2.5}$ sensor and a NO_2 sensor when the drone was not moving but was floating showed a difference in readings. Thus, when the drone was on the ground, the readings using both sensors showed a normal distribution of data [5].

But when the UAV was in the air, the sensor readings changed because of the electronic interference from the UAV. However, the $PM_{2.5}$ concentrations may be compared with those obtained by the reference method [5].

Other researchers are working on drones that can collect water samples. Monitoring of natural and public waterways is improved by drone sampling. The available sensors for in situ water analysis include temperature, conductivity (a proxy for salinity), pH, dissolved oxygen, and oxidation-reduction potential [8].

The drone-mounted sensors used in the design of maps and models for the quality and quantity of water bodies appeared around 2013 [12].

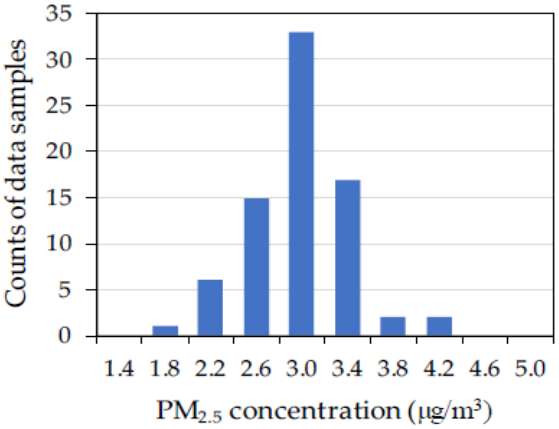

Fig. 6. *$PM_{2.5}$ measured on ground* [5]

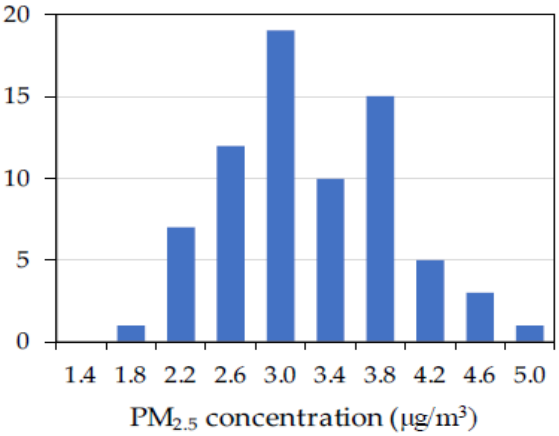

Fig. 7. *$PM_{2.5}$ measured in air* [5]

The widely researched water quality parameters included conductivity, salinity, water reaction pH, chloride Cl-dissolved oxygen DO, total suspended solids (TSS), chlorophyll, turbidity, potassium cation K^+, ammonium nitrogen (NH_4^-N), sodium (Na^+), biological oxygen demand BOD, magnesium (Mg^{2+}), total phosphorous, orthophosphate (PO_4^-P), total nitrogen, iron ions (Fe^{2+}, Fe^{3+}), chemical oxygen demand COD, zinc (Zn^{2+}), calcium (Ca^{2+}), manganese (Mn), copper (Cu), bicarbonate HCO_3^-, cadmium (Cd), chromium (Cr), total coliforms and total hardness. The satellite's remotely sensed data were mostly used to describe the dynamics of the parameters listed above [12].

The studies based on drone remotely sensed data were conducted for mapping and monitoring chlorophyll content and turbidity in lakes, ponds, and dams.

The quality of irrigation water needs continuous monitoring if the aim is to have quality crops and reap rich harvests. COD, conductivity, pH, TSS, DO, and turbidity are essential indicators of water used for irrigation in agriculture [12].

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The drone technologies use sensors for water remote sensing applications, such as multispectral sensors and hyperspectral sensors. The hyperspectral sensors that collect data on water quality by remote sensing are very sensitive to low-intensity changes in water quality parameters such as chlorophyll and TSS concentrations [12].

The multispectral sensors retrieve data from the electromagnetic spectrum at a very high spatial resolution. Thus, those sensors operate in the visible spectrum also red edge and the near-infrared spectrum. In order to monitor the water quality parameters, the predominant working spectrum is the visible (blue and green) and the NIR (near-infrared radiation) wavebands. [12].

A study of water quality from a 1.1 ha agricultural pond using UAV developed a hexacopter designed with an open-source electronic sensors platform that measures the temperature, electrical conductivity (EC), dissolved oxygen (DO), and pH. The measurements collected by an open-source multiprobe meter (OSMM) mounted on the unmanned aerial vehicle (UAV) were compared to the measurements made by a commercial multiprobe meter (CMM) [13].

The commercial multiprobe meter (CMM) contained a portable SENSION meter for measuring pH and EC and a portable HQ10 meter with DO and temperature probes. The OSMM was a combination of a water sensor node and an open-source electronic platform. The water sensor node consisted of EC, DO, pH, and temperature circuits, and was integrated with a microcontroller [13].

Fig. 8. *The open-source multiprobe meter (OSMM) components* [13]

In table 1, the terms mean N: Number, SD: Standard deviation. The differences between the OSMM and CMM measurements for DO, EC, pH, and the temperatures were 2.1 %, 3.43 %, 3.76 %, and <1.0 %. Although EC and temperature values were statistically different, they followed a similar pattern [13].

Table 1. *Descriptive statistics for water quality parameters obtained by the OSMM and CMM* [13]

Quality Parameter	OSMM			CMM			Difference (%)
	N	Mean	SD	N	Mean	SD	
Temp. (°C)	195	27.15	0.93	39	24.79	0.58	2.33
EC (µS/cm)	195	49.2	9.69	39	64.73	4.57	3.43
pH	195	8.43	0.86	39	8.12	0.36	3.76
DO (mg/L)	195	9.05	0.27	39	8.87	0.49	2.08

Soil monitoring is also a current concern in the UAV technologies using sensors.

Research was conducted to obtain the physical properties of the peat soil and to correlate with the healthiness of the pineapple crop. The data was collected from the site using DJI-Phantom 4 drone and Mapir Survey 3 multispectral camera. The peat soil samples were taken and were transferred to the laboratory to be tested for moisture content and pH test [14].

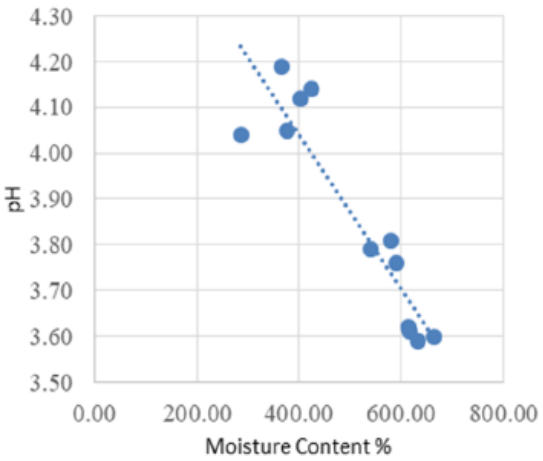

Fig. 9. *PH value and moisture content* [14]

The moisture content of the soil was increasing to 100 % of water ratio, while the value of pH of acidity was decreasing. Both the physical properties of the peat soil were dominating factors contributing to the healthiness of the pineapple crop in the research area [14].

Another study used hyperspectral sensors mounted on a UAV to evaluate and map soil salinity. The quantitative assessment and evaluation of soil salinity at the field-scale level were achieved using electromagnetic induction equipment (EMI) and a hyperspectral camera installed on a UAV platform. The calibration of apparent electrical conductivity (ECa) values measured by the EMI sensor was

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performed by the empirical line method using the lab measured electrical conductivity derived from soil samples. The ECA was measured with the EM38-MK2 sensor by first inducing an electrical current in the soil. In order to correlate the spectral information to soil salinity contents, the random forest (RF) regression was used. An empirical linear regression was established between the ECa and $EC_{1:5}$ [15].

Table 2. *The points values of ECa and the $EC_{1:5}$ measured by hand-hold EM38-MK2 and chemical analysis* [15]

Field	Conductivity	Descriptive Statistics (EC_a, mS m^{-1}; $EC_{1:5}$, dS m^{-1})		
		Min	Max	Mean
A	EC_{ah}	571.15	955.72	765.05
	EC_{av}	598.15	1065.57	846.74
	$EC_{1:5}$	20.25	54.90	37.64
B	EC_{ah}	450.20	1092.15	830.47
	EC_{av}	585.67	1035.90	824.51
	$EC_{1:5}$	7.20	14.68	11.73
C	EC_{ah}	695.86	1126.99	890.15
	EC_{av}	560.17	955.56	778.00
	$EC_{1:5}$	9.64	19.64	14.11

In Table 2, the highest EC value (47.14 dS m^{-1}) of the surface soil was measured in field A which had no vegetation at all. The spectral reflectance of crops on the soil would change due to poor water absorption under conditions of salt stress.

The UAV platform used for establishing regression models between EC and soil surface reflectance turned out to be acceptable for collecting spectral information. In addition, the soil salinity evaluation was more accurate due to results for bare land and sparse vegetation area and less specific for dense vegetation area [15].

An aerial monitoring platform equipped with sensors for detection of specific contaminants, named AWISEM (Air-Water Innovative System for Environmental Monitoring) was aimed to be a mobile aerial and aquatic vehicle that should contain: A) an aerial and aquatic mobile monitoring platform, with an innovative sustentation system, having the capability to fly and also to float in an aquatic environment; B) two distinct sets of sensors, based on CNT (carbon nanotube-polymer) composites with high sensitivity for the monitoring of either aerial or aquatic contaminants. The sensing membranes (thin layers) for two types of sensors are able to detect contaminants such as ammonia - in air and nitrites - in water [16].

3. Conclusions

The digital revolution has the potential to change the way we approach monitoring and sensing important fields. Combining drone technology with high-tech sensors, reliability, dynamics, and efficiency are achieved in applications such as monitoring water bodies, soil fertility, irrigation, planting trees, air pollution, etc.

The UAV system integrates sensors as electromagnetic spectrum sensors, gamma ray sensors, airborne wireless sensor network, biological sensors and chemical sensors, etc. for these sensing and monitoring applications.

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MODELLING OF CORROSION INHIBITION OF CUCUMBER PLANT EXTRACTS ON AISI 1007 STEEL IN SEA WATER

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ABSTRACT

Adsorption Models with the application of corrosion experimental data is a very popular mechanism to predict various inhibitive systems. The effective modelling and interpretation of adsorption isotherms reliably determine the level of accuracy of adsorption processes. This study aims to apply the adsorption models and inhibitive mechanism of Cucumber Peel Extract (CPE) and Cucumber Seed Oil (CSO) to corrosion of AISI 1007 steel grade in the saline medium using both the electrochemical (Tafel Polarisation) and non-electrochemical (Weight Loss) techniques. The chemical composition of AISI 1007 and the phytochemical properties of studied extracts were determined. Consideration was given to Langmuir and Dubinin-Radushkevich Isotherm models (D-RIM) to study the inhibitive properties of CPE and CSO on AISI 1007 steel in an aggressive medium. The result of inhibition efficiency from weight loss measurement showed maximum inhibitions of 94.44 % and 95.44 % with 1.0 g/L concentration of CPE and CSO respectively in sea water medium. The result of the studied extract at 25 ℃ in seawater showed that the corrosion current density of AISI 1007 steel decreased and increased in the inhibition efficiency with 87.33% and 94.67% for CPE and CSO respectively. The negative value of ΔGads was greater than 20 kJ/mol and was obtained as a result of electrostatic interaction between the adsorbed inhibitor molecules and the ions/atoms on the metal surface. The studied inhibitors were confirmed to be mixed organic corrosion inhibitors type. The values of E and maximum surface coverage (θmax) for the two measurements are satisfactorily in acceptable agreement as similar to the range of value obtained for inhibition efficiency.

KEYWORDS: adsorption; mechanism; modelling; cucumber; corrosion

1. Introduction

The use of inhibitors is one of the superlative processes to improve corrosion resistance of metals caused by acid solutions such as in desalination plants as well as in other industrial applications. Recently, efforts have been shifted towards the formulation of safe inhibitors, like plant extracts that have become the reliable eco-friendly, readily available, and renewable sources of effective corrosion inhibitors [1, 2]. The inhibition characteristics of green inhibitors exhibit varying inhibitive properties using different interpretations of adsorption isotherm models. Ayawei *et al.* provided an evaluation of all adsorption isotherm applications using both linear and non-linear regression analysis. In addition to error functions for optimal adsorption analysis and among the review models, the two-parameter isotherm has been

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considered one of the most widely used tools for identifying the best-fitting adsorption models due to its estimation of the distribution of adsorbates throughout the surface coverage, evaluation of the adsorption system, and validation of the consistent quality of the theoretical assumptions of the adsorption isotherm model [3].

In recent time, Langmuir, Freundlich, Dubinin-Radushkevich, Temkin and Flory-Huggins models have been considered for green corrosion inhibition analysis among others [4]. Many researchers reported the use of these isotherm models for modelling of experimental data in predicting the inhibitive mechanisms of plant extracts such as Bitter Leaves Root, Watermelon Rind, Cucumis Sativus (Cucumber), Senecio anteuphorbium Leaves, and Acacia Tortilis Bark and Leaves [4-8]. However, the model used to investigate the mechanism of these green inhibitors on the surface of the metal is acidic media, and the feasibility of experimental techniques for corrosion is also required. Therefore, this study applied two different adsorption models to predict the viability of experimental techniques and the mechanism of corrosion inhibition on AISI 1007 Steel in natural seawater medium using Cucumber plant extract.

2. Methods and materials

2.1. Preparation of Inhibitors

The Cucumber (Cucumis sativus) was obtained locally in Ilorin, Nigeria. To remove unwanted material, the vegetable was thoroughly washed and peeled. The cucumber was cut for a proper view of the seed, and the seeds were carefully removed. The peel and seeds were air-dried at room temperature, and pulverized into 20 µm. The weighed inhibitor was stored in desiccators prior to use. To prevent evaporation, a 20 g ground sample was combined with ethanol and sealed firmly for 48 hours. To obtain a high concentration of the produced extracts, the Cucumber peel Extract (CPE) and Cucumber Seed Oil (CSO) were filtered. The filtered solution was heated in a rotary evaporator for 20 minutes at 78 °C in a rotary evaporator configuration to expel the ethanol.

2.2. Preparation of Specimens

The 1.5 mm thickness of AISI 1007 steel specimens' elemental compositions was determined with spectrometer (Serial number: 15007384) containing 0.015% P, 0.008% S, 0.220% Mn, 0.033% C, 0.034% Si, 0.038% Cr, 0.007% Ni, 0.011% Al, 0.004% Co, 0.021% Cu. The corrosion specimens were prepared according to ASTM G4 standard [9]. The coupon dimensions of 2.2 x 1.7 x 0.15 cm were polished to a mirror finish, degreased with trichloroethylene, and properly labeled for identification with rope, and for easy removal from the environment. Subsequently, the specimens were degreased in ethanol, air-dried in acetone, and stored in desiccators.

2.3. Weight Loss Method

The composition of the used seawater was carried out at the Chemistry Department laboratory, University of Ilorin, Ilorin, Nigeria, presented in Table 1. The preparing, cleaning, and evaluating Corrosion Test specimens were carried out according to ASTM G1 standard [10]. The specimens were weighed with an electronic weighing machine (HX 302T) with an accuracy of ±0.01 g to establish the initial weight of the specimens (W1) and subsequent measurements.

Table 1. *The composition of seawater*

Composition	Value
pH Value	8.90
Electrical Conductivity	123.50
Turbidity (NTU)	10.42
Chloride ion (mg/l)	15.20
NaCl (mg/l)	63.7
Na ion (mg/l)	48.50

The immersion corrosion testing of specimens was conducted according to ASTM G31 standard [11]. The prepared steel specimens were immersed in 750 mL of seawater with and without varying concentrations of Cucumber extract (Peel and Seed oil). The concentrations of the cucumber extracts (peel and seed oil) were altered from 0.1 g/L to 1.0 g/L. All the test specimens were removed from the solutions every 24 hours for consecutive 3 days and 168 hours intervals for 1 month then the specimens were air-dried. The dried specimen was then reweighed to determine the difference in the weight of the specimens. The procedures were repeated for 24 hours intervals for the first 3 days and 168 hours intervals for 14, 21, and 28 days of exposure. From the weight loss, the Corrosion rate and Inhibiting efficiency (IE %) and surface coverage of the plant extracts were determined using Equation 1 [2]:

$$\text{Corrosion rate (mpy)} = \frac{534W}{DAT} \quad (1)$$

W is the change in weight of the specimen (g), D = 7.85 g/cm^3, the specimen's constant density; A is the surface area of the AISI 1007 steel coupon (cm^2),

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while (T) is the exposure period in (hour). The percentage inhibiting efficiency (IE %) using the Equation 2 [4]:

$$I.E\ (\%) = \frac{CR_{Black} - CR_{inh}}{CR_{Black}} X100 \quad (2)$$

where: CR_{Black} = Corrosion rate in the absence of inhibitor, CR_{inh} = Corrosion in the presence of inhibitor.

2.4. Electrochemical Measurement

Electrochemical studies shown in Figure 1 were carried out at room temperature using a system with three electrode cells having AISI 1007 steel samples as working electrodes (having a 1.0 cm^2 exposed area), a saturated calomel electrode as the reference electrode, and a graphite rod as the auxiliary/counter electrode. The exposed area (1.0 cm^2) of the working electrode (AISI 1007 Steel) was polished with a series of emery sheets of variable grades and washed thoroughly with double distilled water prior to immersion in the cell. During the polarization experiment, 0.01 V s^{-1} was taken as the scan rate, 0.0 s hold time at Ef, and 2.0 s as quiet time. Potentiostat (VersaSTAT) was used as an electrochemical workstation. The workstation is embedded with software including DC105 for the TAFEL Polarisation. The experimental data was obtained for data analysis. The Tafel polarization curve showed the called Tafel behavior, which is the linear behavior in the E against log (i) plots. The Ecorr and i_{corr} were determined by extrapolation of the slope in the polarization curves back to the corrosion potential.

The corrosion current density i_{corr} or corrosion rate is represented by the point of intersection, and the related potential at that point is Ecorr. The corrosion rate (MPY) used for potentiodynamic polarization is given by Popov [12]:

$$CR\ (MPY) = \frac{0.131\ X\ I_{corr}\ X\ EW}{d} \quad (3)$$

where d = 7.874 kg/m^3 (iron metal's density), and EW is the equivalent weight per valence state of iron metal in oxidized form of 2 is 27.92.

The Inhibition efficiency (% IE.) of AISI steel samples was computed based on corrosion current density measurements of inhibited AISI steel electrode (i_{corr}) using the following relationship [8]:

$$IE\ (\%) = \frac{i_0 - i_a}{i_a}\ X\ 100 \quad (4)$$

where i_a is the corrosion current for the control sample, and i_0 is the corrosion current for the inhibited samples at different concentrations.

Fig. 1. *TAFEL Setup*

2.5. Adsorption Models

The Langmuir isotherm model was considered to investigate the mechanism of the interaction between the inhibitor and the AISI 1007 steel surface. The effect of surface coverage (θ) was determined for different concentrations of the studied extracts (inhibitors) from weight loss and electrochemical measurements with expression IE % = θ X100 as reported by Awe *et al*. [4]. The equilibrium constant (K_{ads}) of the adsorption process was obtained from the isotherm model [8] as follows:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

where C is the concentration and θ is the surface coverage.

Values of equilibrium adsorption constant (K_{ads}) obtained determined from the intercept of Equation 4 and related to obtain free energy of adsorption (ΔGads) in Equation 6:

$$\Delta G_{ads} = -RT \ln (999\ K_{ads}) \quad (6)$$

where R is the molar gas constant, T is the room temperature of adsorption and 999 is the molar heat of adsorption of sea water.

Corrosion experimental data achieved was also employed in the Dubinin-Radushkevich Isotherm model (D-RIM). The model explains the mechanism of adsorption of corrosion inhibition and the capability of experimental methods for corrosion. The mathematical model for D-RIM with the maximum surface exposure (θ_{max}) is as follows in equation 7 [3] and the polany potential (δ) is given in Equation (8):

$$Lin\theta = lin\theta_{max} - a\delta^2 \quad (7)$$

$$\delta = RT \ln (1 + \frac{1}{C_{lin}}) \quad (8)$$

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From the equation, the inhibitor concentration has an absolute temperature (T) and the molar gas constant (R). The constant (a) represents the mean adsorption energy (E) stated in equation (9). This is the transfer energy of 1 mol of adsorbate from infinity (bulk solution) to the adsorbent's surface.

$$E = \frac{1}{\sqrt{2a}} \quad (9)$$

3. Results and discussion

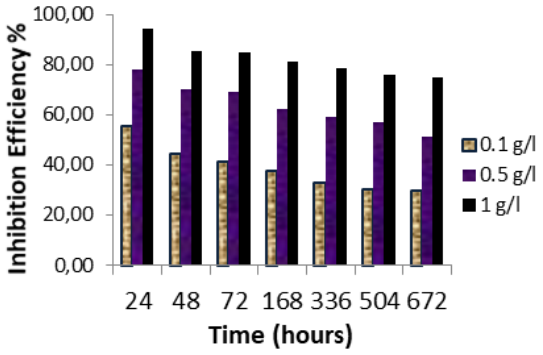
3.1. *Weight Loss Measurement*

The various corrosion parameters are shown in Table 2 include corrosion rate, surface coverage, and inhibition efficiency that were obtained. On the metal surface, prior to the beginning of the adsorption process of the inhibitor, the corrosion rate was significantly high initially, with little or no inhibitive impact from the extract, regardless of inhibitor concentrate. The corrosion rates decreased with an increase in concentrations of the inhibitor (CPE and CSO) and the lowest values of 0.0066 and 0.0054 MPY for CPE and CSO respectively) were obtained by applying the highest concentration (1.0 g/L) of the studied extracts. The values of inhibition efficiency (IE%) and that of surface coverage (θ) for the investigated inhibitors were observed to rise with the increase in inhibitors concentrations, which might be the formation of a passive layer period in the solution. Dimeric film formation on the surface of mild steel right from the period of passive layers formation in the solution resulted in some non-uniformity in values of inhibition efficiency [4].

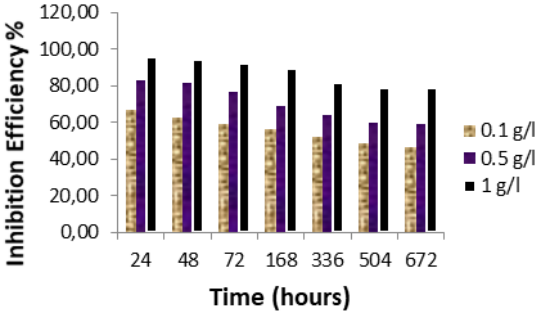
Table 2. *The Corrosion Parameters for Inhibition of AISI 1007 steel in seawater*

Inhibitors	Concentrations	Corrosion Rate	(IE%)	Θ
Blank	-	0.1180	-	-
CPE	0.1	0.0524	55.56	0.5556
	0.5	0.0262	77.78	0.7778
	1.0	0.0066	94.44	0.9444
CSO	0.1	0.0393	66.67	0.6667
	0.5	0.0197	83.33	0.8333
	1.0	0.0054	95.44	0.9544

For the studied inhibitors, the IE% was displayed in Figure 2. It can be observed that IE% decreases as immersion time increases. The highest inhibition was recorded at 24 h of immersion as 94.44 % and 95.44 % for CPE and CSO respectively. When the immersion time was increased, a clear decrease was observed. These results may be due to the instability of the inhibitor layer or due to the biodegradable nature of the plant's extracts after long-time adsorption [8].



(a) CPE Inhibitor



(b) CSO Inhibitor

Fig. 2. *Plot of Inhibition Efficiency against Exposure Time for Study Inhibitors*

3.2. Electrochemical Measurement

The electrochemical parameters related to Tafel plots like corrosion potential (E_{corr}), corrosion current density (i_{corr}), corrosion potential (E_{corr}), and the corresponding IE% for the corrosion of AISI 1007 steel in seawater solution in the absence and presence of different concentrations (0.1-1.0 g/L) of CPE and CSO at 25 ℃ are illustrated in Table 3. The results of the inhibited metal specimens in seawater revealed lower i_{corr} values and more positive corrosion potential (E_{corr}) compared to As-received AISI 1007 steel. This suggests that the studied CPE and CSO protect the metal against corrosion through the effective stability of the passive layer.

The value of i_{corr} for 0.1 g/L concentration of studied extracts on the sample was 4.0E-09 A/cm^2 at 2 hours exposure time which was reduced to 2.47E-09 A/cm^2 in the case of 0.5 g/L CPE, and 1.14E-09 A/cm^2 was obtained for 1.0 g/L concentration of CPE. The value of i_{corr} was found to decrease on increasing the concentration of the inhibitors in sea water.

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Table 3. *Potentiodynamic polarization parameters for corrosion Inhibition of AISI 1007 steel using CPE and CSO in Saline water*

Inhibitors	Concentrations	Ecorr (mV)	I_{corr} (Acm^{-2})	IE.%	CR (mpy)
Blank	-	-	9.0E-09		4.18E-09
	0.1	0.296	4.0E-09	55.56	1.858E-09
CPE	0.5	0.299	2.47E-09	72.56	1.147E-09
	1.0	0.302	1.14E-09	87.33	5.295E-10
	0.1	0.26	3.42.0E-09	62.00	1.588E-09
CSO	0.5	0.281	1.55E-09	82.78	7.20E-10
	1.0	0.292	0.48E-09	94.67	2.230E-10

The result in sea water of the studied extract at 25 ℃ revealed that the corrosion current density of AISI 1007 steel decreased and the corrosion inhibition efficiency increased to 87.33% and 94.67% for CPE and CSO respectively. Results also suggest that the addition of 1.0 g/L concentration of studied extract in seawater improves the corrosion inhibitive performance of metal. While the lower i_{corr} values showed the formation of inhibitive strength on the metal surface.

However, the anodic and cathodic parameters of potentiodynamic polarization curves for the tests are shown in Figure 3. The anodic and cathodic current densities as shown in the potentiodynamic polarization curves for the studied inhibitors decreased and this can be explained by the adsorption of organic compounds such as heteroatoms (nitrogen-oxygen, phosphorus, and sulphur) at the AISI 1007 steel surface. The aromatic rings found in organic compounds acted as mixed-type inhibitors. This behavior blocked the reaction sites on the metal surface which is due to the good coverage of the metal surface by inhibitor molecules [1, 3].

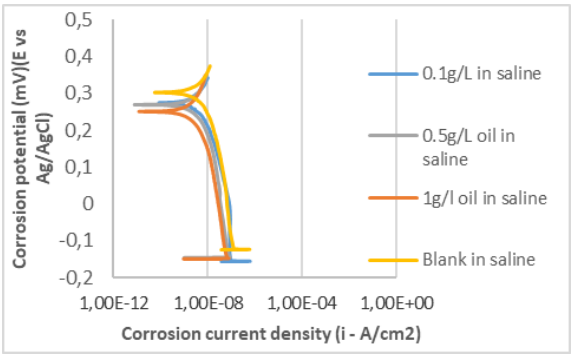

Fig. 3. *Corrosion Potential against Corrosion Current Density for CSO Inhibitor*

3.3. Adsorption Properties of Corrosion Inhibition Langmuir Adsorption Model

The negative values of free energy of adsorption, ΔGads, are obtained (Table 4), the negative value of ΔGads implies a spontaneous adsorption process and the excellent stability of the adsorbed layer on the AISI 1007 steel surface. The value of -ve ΔGads is greater than 20 kJ/mol that involves an electrostatic interaction between the atoms/ions on the metal surface and the strong adsorbed molecules [1].

Table 4. *Langmuir Adsorption Model Parameters*

Inhibitors	Methods	K_{ads}	ΔG_{ads}	R2
CPE	WL	9.1239	-22.62	0.9955
	PDP	9.7217	-22.77	0.9954
CSO	WL	14.3916	-23.75	0.9978
	PDP	12.6922	-23.44	0.9983

The result of mixed adsorption obtained involves physical and chemical adsorption. Generally, the values of ΔGads decrement to -20 kJ/mol are consistent with electrostatic interaction between the carbon steel surface and charged molecules of inhibitor (physical adsorption) and those higher than -40 kJ/mol involves transfer or sharing of charge from the molecule inhibitor to a steel surface in order to form a coordinate bond (chemisorption).

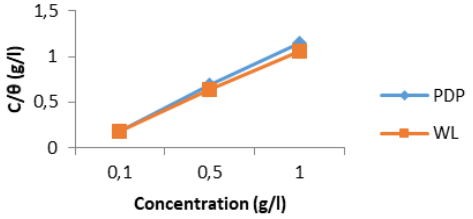

Fig. 4. *Plot of Langmuir Adsorption Model*

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3.4. Dubrimim–Radushkevich Adsorption Model

The type of adsorption was achieved from the information given based on the magnitude of E and once the values are less than 8.0 $KJmol^{-1}$, its shows physical adsorption [14]. Figure 5 showed the interaction of ln θ for the data obtained from the different methods.

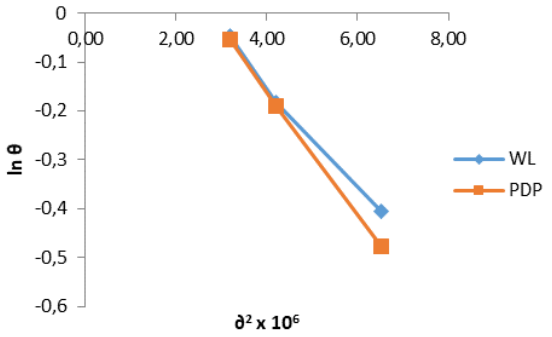

Fig. 5. *Plot of D-RIM for Corrosion Inhibition of AISI 1007 Steel in seawater*

From the D-RIM, the parameters are stated in Table 5, which reveals the values of energy transfer (E) that indicated the physical adsorption mechanism in which the values of maximum surface coverage (θ_{max}) for each of the methods used in this study are in good agreement as similar to the variability of the inhibition efficiency values obtained.

Polyphenols, including phenolic compounds and flavonoids, constitute the main groups of compounds acting as primary antioxidants or free radical scavenges. In the plants, a wide variety of free radicals scavenging molecules that are rich in antioxidant activity such as alkaloids, flavonoids, saponin, tannin, and terpenoids are present [15]. Their antioxidant activity is based on their ability to donate hydrogen atoms to free radicals and their stability as radical intermediates.

It can be observed from the obtained result that both plant extracts have various phytochemical compounds which are presented in Table 6. These extracted compounds chemically bind to AISI 1007 steel by scavenging the free-oxygen radicals that produce a relatively stable radical. The free radicals' metastable chemical entities have a tendency to capture electrons from molecules in their instant local vicinity [16]. This resulted in the creation of a barrier formation on the surface of the specimens in the corrosive attack. It can be observed that both extracts have high flavonoids.

Table 5. *The D-RIM Adsorption Parameters for Corrosion Inhibition of AISI 1007 Steel in Seawater*

Inhibitors	Methods	θ_{max}	a	E	R^2
CPE	Weight loss	15.78	0.1574	6.35	0.97
	PDP	13.91	0.1522	6.56	0.95
CSO	Weight loss	17.47	0.177	5.65	0.96
	PDP	16.78	0.1682	5.94	0.98

Table 6. *Phytochemical analysis of Cucumber Peel and Seed oil*

Parameter	Cucumber Seed oil	Cucumber Peel
Alkaloids	1.32 (+)	0.89 (+)
Flavonoids	2.86 (+)	2.72 (+)
Tannis	1.16 (+)	0.15 (+)
Saponins	0.09 (+)	0.06 (+)
Terpinoids	0.13 (+)	0.07 (+)
Phenols	0.28 (+)	0.20 (+)
Cardiac glycoside	0.09 (+)	0.08 (+)
Steroids	1.38 (+)	1.32 (+)
Phobatannins	0.18 (+)	0.14 (+)

The presence of a large quantity of flavonoids in cucumber fruit homogenate indicates the ability to scavenge free radicals as it was reported as the chief source of antioxidant in plants that have been known to play a significant role in free radical scavenging [17].

4. Conclusions

The corrosion inhibition was conducted using weight loss, and electrochemical measurements. CSO displays good inhibition efficiency, while CPE shows a moderate inhibition performance. The inhibition properties revealed that both CPE and CSO mitigate both the anodic and cathodic process (mixed-typed inhibitor) following Langmuir and Dubinin-Radushkevich Isotherm adsorption model. Results showed that the performance of both inhibitors decreased with time. The study showed that both plant extracts contained different phytochemical

compounds which in the adsorption process created a barrier formation on the surface of AISI 1007 steel in the corrosive attack.

Acknowledgments

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IMPACTS OF COVID-19 PANDEMIC ON NIGERIA'S ECONOMY: EFFECTIVE FOUNDRY TECHNOLOGY AS A SUSTAINABLE SOLUTION

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ABSTRACT

This study considered the effect of the Covid-19 pandemic on the economy of Nigeria and investigated the effective application of foundry technology to proffer a sustainable solution to the problem. Domestication of production of motorcycle components, being a significant means of transportation that is required for the development of any society was considered. Aluminium scraps from a waste dump and Belle and Oyun natural moulding sands in Kwara State, Nigeria, which have been previously recommended for non-ferrous casting in previous studies were considered and used for casting of motorcycle/tricycle parts (centre stand and brake pedal) using AFS guidelines. The scraps' chemical compositions were analysed. Thereafter, some mechanical properties with the microstructure of the aluminium casts and that of commercially obtained imported types were examined using appropriate ASTM standards. Cast aluminium alloy produced using scrap had no casting defects and had some mechanical properties comparable to those available on the markets (imported). Thus, effective usage of naturally available moulding sands in Nigeria for foundry applications would help to proffer sustainable solutions to the nation's economic problem as a result of the adverse effects of the Covid-19 pandemic.

KEYWORDS: Covid-19, Aluminium alloy, Casting, Foundry, Moulding sand

1. Introduction

The effects of the Coronavirus pandemic (Covid-19) in combination with a decline in the oil price are generating Nigeria's financial and economic collapse (economic crises or recessions) [1-4]. According to Saliu (2021), the recent Nigerian economic recession is the worst recorded in almost four decades by the West African nation [1]. The economy of Nigeria, which had earlier been fragile is being crumbled and traumatic as a result of the sharp decline in productivity, jobs, and income due to the Covid-19 pandemic lockdown [3, 4]. The economic activities have been destabilized due to disruption in the supply chain across sectors, collapse in capital flight, commodity prices, and turmoil in the capital market during and after the lockdown.

National Bureau of Statistics (NBS) data (as of September 2020) revealed that Nigeria's trade stood at -14.95% as of the second quarter of the year 2020 with a decrease of -17.97% if compared with that of the year 2019. The nominal gross domestic product (GDP) growth in manufacturing was 0.14%, which was -37.92% lower than that of 2019; the agricultural sector recorded a decrease of -0.21% and -0.62% compared to that of 2019 and 2018 respectively [5].

The oil and gas sector, which is the main source of revenue for the Nigerian government has also been significantly affected by the spread of the Covid-19

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pandemic. The oil prices rapidly decreased in value by 60%. Thus, the Nigerian economy is expected to be weakening as a result of the pandemic [6]. In the year 2020, negative cumulative GDP (-2.48%) was recorded for the first nine months [1]. The National Bureau of Statistics revealed that negative growth was recorded for two consecutive quarters in Nigeria in the year 2020. NBS reports have shown that the results of COVID-19 with a significant low volume of exports rendered over 21 million Nigerians unemployed (27% of the labor force) and such resulted in Nigeria's economy being contracted by 6.1% year on year in the second quarter of the year 2020, which is the steepest in the last ten years [7].

Nigeria as a country needs to rapidly embark on the economic diversification of its economy from oil and gas, which can be achieved through local production of goods (like engineering products, tools and devices, vehicle parts, among others) and effective utilization of its resources. This will also help in the establishment and sustainability of Small and Medium Scale Enterprises (SMSE) that are required to provide a sustainable solution to the problem of the economic downturn due to the lockdown as a result of the COVID-19 pandemic and the issue of unemployment among the nation's youth [8]. Industrialization is an essential factor in achieving Nigeria's desired development [9,10]. Production capacity from the availability of raw materials to the technology used in transformation of the raw materials to finished products determines the success of industrialization [9].

For the development of any society, transportation is significant as it facilitates community development, optimum utilization of resources, allows and enhances the movement of people, and offers accessibility to hitherto inaccessible areas easy [11]. The economic development of many nations could be hindered due to a lack of transport facilities since transportation forms the basis of all socio-economic interaction [12, 13].

The three main modes of transportation in Nigeria are road, air, and marine. Road dominated the means of transportation in urban areas of Nigeria [14]. Road transport in Nigeria is said to account for more than 90% of the movement of goods and passengers in the country as of now [12]. Ezeife and Bolade (1984) revealed that the road as a mean of transportation had the greatest influence on the socio-economic development of Nigeria [15]. Road transportation is the most prevalent and effective form of transportation in Nigeria [12, 13, 16, 17].

Road transportation in Nigeria involves the use of bicycles, tricycles, motorcycles, cars, buses, and different types of trucks among others. Recently, the use of tricycles and motorcycles for transporting goods and services for private and commercial purposes is prevalent in Nigeria, due to population rise, unemployment and the lack of an effective urban transport infrastructure scheme [11]. The collapse of the public intra-city transport system is another factor that increased the use of motorcycles in commercial transportation by about 70% and this sector is dominated by private entities [14, 18]. One of the various means of transportation currently available in this country is the tricycle (KEKE NAPEP) which is also a source of empowerment for the youth and a source of living for a large number of people in this country [19].

Through several studies of motorcycles and tricycles usage in public transportation for commercial or personal uses in Nigeria, it was discovered that a decrease in the supply of new vehicles, economic depression, rapid rate of urbanization with inadequate transport facilities, unemployment, relative lucrative nature of using motorcycle and tricycles or bicycle for transportation, enhance mobility and accessibility to areas with bad road network or where there is traffic congestion, low fuel consumption, fastness, reliability and flexibility, a government programme in eradicating poverty in the country are significant factors that contributed their usage in the country [11, 14, 20-23]. Brands of the motorcycle were diffused into the Nigerian market as an initiative of the Federal Government in the year 2002 to ease transportation problems and create an avenue for self-employment for the unemployed and the jobless Nigerians [24]. For transportation in recent times, the use of paratransit (motorcycles and tricycles) is popular and becoming a mean of full public transport in some areas of the country, due to the failing mass transit infrastructure and road network [25]. Low initial cost, low maintenance cost, ease in modifying, reliability, and fuel efficiency contribute to a rapid increase in their use for commercial purposes [25-28].

The demand for motorcycles / tricycles and their components (parts) in Nigerian markets is tremendously high. The components (such as brake pedal, stand, clutch handle, etc.) need replacement due to failure in service. The motorcycles and tricycles with their components are imported to Nigeria mostly from Hong Kong (China), Japan, and the United States of America and some of these motorcycle parts are manufactured and produced using materials like aluminium alloys or metals. Cast-iron, mild steel, aluminium, composite materials, and polymeric materials were employed in the manufacture and production of brake pedals [29-33]. The brake pedal is one of the major parts of the tricycle, which are imported into Nigeria over the year that normally fails [19]. The center stand is used to prevent the loading of the tires of the motorcycle

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when maintenance works are required on a motorcycle to prevent leakage [27].

The appropriate use of a country's foundry technology plays an important role in the development of the nation's industrial sector development which in turn contributes to its economic growth [34]. Production of castings is essential for the development of every nation in transportation, farming, construction, and mining [35]. Among all foundry materials for casting, sand has been found to be very important because sand is abundant, cheap, readily available, and suitable for casting all kinds of metal [36]. In metal casting manufacturing processes, sand casting is extensively used since almost all metals can be produced by sand casting.

In the manufacturing of metallic engineering wares, vehicle parts, tools, devices, and equipment such as engine blocks, valves, cylinder heads, pump cases, and machine tool bases, metal casting is found suitable [37]. Foundry technology is appropriate for all metals of different sizes and sand cast is found suitable [38, 39]. In the present manufacturing process, the sand-casting process is the most commonly used in metal casting [39]. In the foundry, moulding sand is an important material [40], as the properties of the sand used in casting, largely determine the quality of the cast products [34, 41-44].

In Nigeria, moulding sands have been said to be available but underutilized due to a lack of information about their properties [45, 46]. Studies have revealed that Nigeria is endowed with natural resources like foundry sands which were available and scattered all over the towns and villages in Nigeria in large quantities [9, 34, 47]. These resources (sands) have been used for the production of expensive high-performance engineering equipment; the casting of aluminium cooking utensils, decorative ornament and others for these last decades [34, 47]. Most foundries in Nigeria embark on sand casting without appropriate properties information of the sands used for casting, which will affect the physical, chemical and mechanical properties of the cast [48]. Proper characterization of moulding sand will help eradicate desirable fixtures and produce quality cast. Belle and Oyun natural moulding sand abundantly available in Kwara State have been characterized and found suitable for foundry applications [45, 46].

Aluminium scraps are now becoming more available as a result of an increase in the use of aluminium in many industry sectors especially the automotive and packaging sector [49]. In the automobile sector, aluminium is used in the production of parts, these parts have their service life which is then replaced with another. After which the failed aluminium parts become scrap. While in the packaging sector, aluminium has found increased use as foils and cans due to its chemical inertness, after the products are used [49]. Manufacturing of parts from aluminium scrap requires about 95% less energy than the production of aluminium from its ore [50]. Using these scraps in re-manufacturing aluminium utensils, automobile parts and packaging items will help improve the economy of the country. Hence, proper utilization of the abundantly available moulding sand and aluminium scraps in the production of some of the motorcycle parts will help in reducing the level of importation, reduce the level of unemployment and in turn help the nation's economic growth and development. Therefore, in this study application of some locally sourced moulding sands and aluminium scraps for the production of motorcycle parts through foundry application to proffer a sustainable solution to the effect of Covid-19 on Nigeria's economy through industrialization. According to Bala *et al.*, industrialization serves as the only means that Nigeria aims to become one of the twenty most developed nations [51].

The production of the motorcycle's components can be domesticated in Nigeria due to the abundant availability of the required mineral resources needed in the processes of such parts, such as moulding sand and aluminium. Nigeria is well blessed with viable moulding sands that can be used to cast both ferrous and non-ferrous metals [45, 46].

This paper provides an overview and suitability of available natural moulding sands in Belle and Oyun communities in the Kwara State of Nigeria, which have been characterized [45, 46] for the casting of tricycle brake pad and motorcycle centre stand using aluminium scraps. Successful application of the moulding sands to produce the motorcycle stand and tricycle brake pedal will help to reduce the level of importation of such cycle parts into Nigeria and help to curtail the effect of the Covid-19 pandemic on Nigeria's economy by facilitating their local production and a long-run boost for the economic growth of the country.

2. Materials and methods

2.1. Materials

Samples of the natural moulding sands used in this study were collected from the Belle community, near the river Niger in Bacita and Oyun river bank in Ilorin East of Kwara State. The American Foundry Standard (AFS) for moulding sand sample collection and preparation procedures were adopted in collecting and preparing the moulding sands' samples for the study (Details in [45, 46]). Figures 1 (a and b) show

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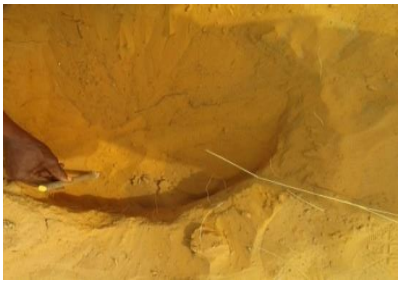
the deposits where the natural moulding sands were obtained.



a

b

Fig. 1. *(a) Belle natural moulding sand deposit (b) Oyun natural moulding sand deposit*

Fig. 2. *Samples of the Aluminium scraps*

The aluminium scraps (Figure 2) used for the casting were procured from a Metal Scraps Seller at Popo-Igbona Area, Ilorin, Nigeria. The aluminium scraps of 10 kilograms were purchased. Commercially available PVC pipe of 20 mm diameter was also obtained and cut to a length of 110 mm and used in the preparation of the pattern for the casting of the specimen.

2.2. Methods

The elemental chemical compositions of the Belle and Oyun natural moulding sands were carried out using X-Ray Fluorescence (XRF) Analyser (Shimadzu 720, Shimadzu Cooperation, United Kingdom made) at Materials Testing Laboratory, University of Lagos, Nigeria in accordance with America Foundrymen's Society (AFS) recommendation in line with practices of earlier researchers as earlier described in previous studies [45, 46, 52].

The properties of the moulding sands, such as the grain fineness number, permeability, refractoriness, green compressive strength, dry compressive strength, specific gravity, moisture content, clay content, and flowability were determined at the Soil Testing Laboratory, Department of Civil Engineering, The Federal Polytechnic, Ado-Ekiti, Ekiti State, Nigeria. The procedures used were in accordance with the guidelines in AFS Standards for the determination of these properties as previously discussed in Shuaib-Babata et al. [45, 46].

Determination of the chemical compositions of the Aluminium scraps used in this study was carried out using Positive Material Identification (PMI) on Olympus Delta Professional (serial number: 5407234) at Midwal Engineering Services Limited, Lekki, Lagos State, Nigeria. The test was done using the principle of Energy Dispersive X-Ray Spectroscopy (EDS). The sample specimen surface was ground and smoothened to remove any debris on the surface and give a very fine-smooth surface, thereafter analysed with the use of a computer system interface.

The steps in the production of the sand cast are illustrated in Figure 3.

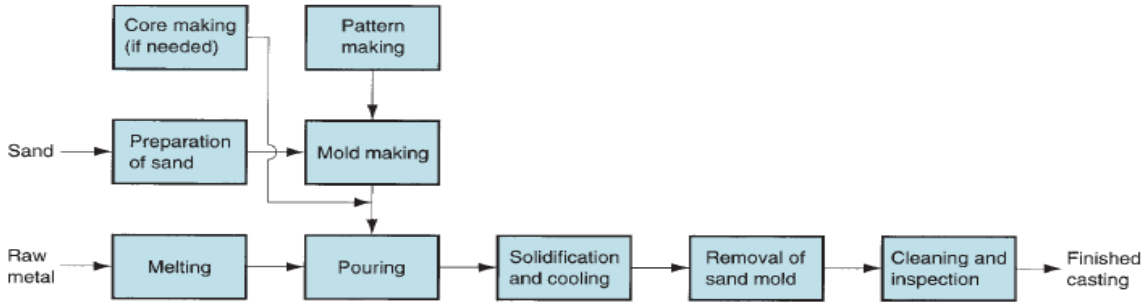


Fig. 3. *Production sequence for sand casting* [53]

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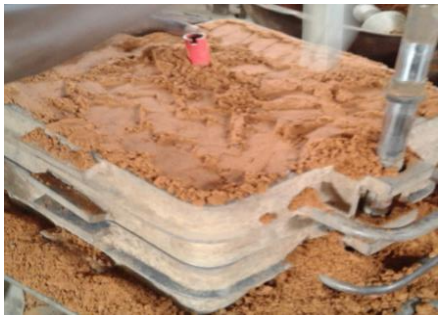
Each of Oyun and Belle natural moulding sand was separately sieved and used in preparing a mould, having the desired motorcycle centre stand and tricycle brake pedal cavity respectively. A sample of each of the natural moulding sands was then thoroughly washed and sun-dried to remove the excess moisture. Thereafter, each sand sample was mixed with water in separate containers. Two full head pan of silica sand was mixed together with one litre of water to produce adequate bonding of the mixture which yield the strength required of the sand. Thereafter, a mould was prepared with this sand mixture along with the PVC pattern to form the specimens' cavities in the mould. The mould contained 5 cavities for the specimens (5 pieces) used to carry out the necessary testing of the final cast. Figures 4-6 show some of the mould making processes.

a

b

Fig. 4. *(a) Sample of Oyun natural moulding sand (b) Sample of Belle natural moulding sand*

a



b

Fig. 5. *After filling the mould with the moulding sand*

The aluminium scrap was melted into a molten metal stage in a crucible furnace at the Foundry Workshop of the Materials and Metallurgical Engineering Department, University of Ilorin, Nigeria. It was allowed to melt at about 700 ºC which is above its normal melting temperature to minimise the drop in the molten metal temperature that would occur as a result of transporting the molten metal from the furnace to the mould cavity. The molten aluminium was poured into the prepared moulds and left to solidify and thereafter, the castings were removed from the moulds. Excess materials in the castings were machined to attain precision. The major common commercially available (imported) tricycle brake pedal models are shown in Figure 7, while details of the casting processes are shown in Figures 8-17.

(a) Bajaj tricycle model brake pedal

(b) TVS tricycle model brake pedal

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(c) Samples of imported TVS tricycle brake pedal with the wooden pattern made

Fig. 7. *The major common tricycle brake pedal models in Nigeria*

Fig. 8. *Wooden patterns used to produce the casts (tricycle brake pedal and motorcycle centre stand)*

Fig. 9. *Cavity of the motorcycle centre stand and tricycle pedal patterns in the mould*

Fig. 10. *The cope and drag after removal of patterns*

Fig. 11. *The moulds ready for pouring*

Fig. 12. *Pouring of the molten metal in to the specimen mould and knocking out operation*

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Fig. 13. *Solidification of the specimen in the mould*

Fig. 14. *The cast specimens before machining*

Fig. 15. *After removing the excess material from the cast specimens (machined casts)*

Fig. 16. *The produced casts (tricycle brake pedal and motorcycle stand)*

Fig. 17. *The cast specimens from the mould for various tests*

The cast products and that of the commercially obtained tricycle brake pedal and motorcycle centre stand were evaluated for tensile, impact, and hardness properties. The tensile and hardness tests were carried out at National Centre for Agricultural Mechanization (NCAM), Ilorin, Nigeria using the Testometric Materials Testing Machines (UTM) (Model No 0500-10080, Win test analysis; 100 KN capacity, England made).

The specimens from each of the casts using Belle and Oyun natural moulding sands for the tensile test were machined and tested following procedures in America Standard Test for Materials ASTM E8/E8M and ASTM, B 557M – 02a standard [54, 55] for dimension and procedures for tensile testing of non-ferrous metals as a guide. Figure 17 shows both (a) diagrammatical representation of the tensile samples with dimension, (b) samples of the specimens machined for the tensile test, and (c) samples of the specimens after the test.

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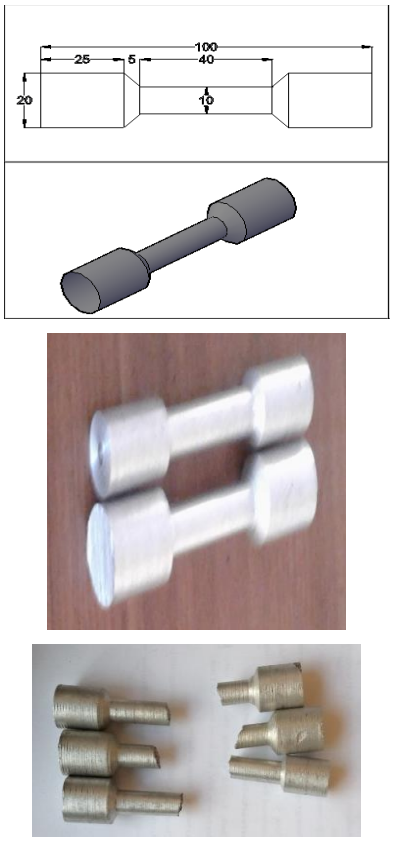

Fig. 18. *Samples of the specimens used for the tensile test (All dimensions are in mm)*

Each of the specimens used for hardness tests was machined to 6.0 mm in length and 20 mm in diameter. The specimens' surface was ground to remove any debris or impurities on the surfaces. On the UTM, a hardened indenting steel ball was pressed between 10 to 15 seconds to indent the specimen to a specific depth into the surface of the specimen but with a different gripping device from that of the tensile test. After removing the ball and load, the diameter of the indentations was measured and input into the UTM. The Brinell Hardness Number (BHN) was automatically determined and obtained as displayed on the computer screen attached to the Testometric machine. Samples of the specimens (a) before the test and (b) after the test (with indentation) are shown in Figure 19.

Fig. 19. *Specimens for the hardness tests*

The impact test was carried out at the Department of Mechanical Engineering, Faculty of Engineering and Technology, University of Ilorin, Ilorin, Nigeria using the Charpy method. The specimen was machined to ASTM standard of 2.0 x 2.0 mm for V-notch. The specimen was held unto the machine and a pendulum was released from a specific height to hit and deform the specimen and the sudden energy absorbed by the specimen was recorded directly from the gauge on the machine. This was done using the methods specified in ASTM E23-18 [56] and E2298-18 [57]. Figure 20 shows (a) the impact testing machine with one of the specimens for the impact test and (b) some specimens after the test.

a

b

Fig. 20. *The impact testing machine and samples of specimens for impact test*

The microstructures of the aluminium cast products were determined through microstructural analysis of the cast specimens. The test was carried out at Engineering Materials Development Institute (EMDI), Akure, Ondo State, Nigeria. The cast aluminium test piece was cut and polished to a good smooth surface using a Belt Grinder with fine grades of abrasive papers to ensure a better surface finish. The aluminium test specimen was then washed in warm water, rinsed thoroughly and allowed to dry. Etching was done using a chemical reagent (Keller's reagent). After which it was allowed to dry before placing the specimen under the microscope (OMAX microscope) with the specification of 14 MP to view and record the microstructure of the surface at a magnification of x100 µm, x400 µm and x 1000 µm.

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Thereafter, the surface was observed with the use of an optical microscope.

3. Results and discussions

The chemical constituents present in the sands are displayed in Table 1. Silica (SiO_2) constitutes the main element in the sands, with 92.93% and 83.79% weight for Belle and Oyun natural moulding sand, followed by Alumina (Al_2O_3) which accounted for 4.8% (Belle) and 11.2% (Oyun sand). The sands were found to be of high-quality silica sand. Thus, the sands belong to the Alumina-silicate group of sand. Since the sands were found to be silica sand through their chemical compositions values which were within the AFS acceptable range values for moulding sands, thus it was recommended to be suitable for casting of metals, most especially metals with low melting temperatures [45, 46].

Silica is very important in moulding sand because it determines the sand refractoriness and chemical inertness. A higher percentage of silica shows the higher refractoriness of the sand [58], which serves as an indication of the likeness of its good refractoriness. High quality silica sand with uniform physical characteristics is the foundry sand for metal casting [38].

The Fe_2O_3 contents in Oyun sand are reflected in its fairly brownish sand colour with a sub-angular shape. Oyun sand had no ZnO which is one of the significant constituents of the clay content in the sand [45]**.** Thus, it may have little effect on the strength of the sand because clay serves as a binder in the moulding sand which holds the grains of the sand together [59]. However, this effect can be eliminated through the addition of a binder and some other additives to the sand [60]. Likely suitability of the two moulding sands for sand casting of both ferrous and non-ferrous metals, especially aluminium through chemical analysis and the likely need for improvement of Belle sand before use had been earlier revealed by Shuaib-Babata *et al.* [39, 40].

Table 1. *Elemental composition of the moulding sand* [45, 46]

S/N	Constituents	Weight (%)		Recommended values for moulding sand
		Belle	Oyun	
1	SiO_2	92.93	84.58	80 to 90% [61]
2	Al_2O_3	5.130	7.22	4-8% [61]
3	CaO	0.010	0.21	
4	MgO	0.068	0.20	
5	Na_2O	0.780	2.06	
6	Fe_2O_3	0.030	4.06	2-5% [61]
7	K_2O	0.385	1.45	
8	MnO	0.006	0.02	
9	ZnO	0.300	-	

The levels of silica, alumina and iron oxide were within the standard specified values for moulding sands [61]. The presence of any of CaO, K_2O, and NaO_2 in moulding sand serves impurities which lowers the refractoriness [58] and the suitability of the sand for casting of ferrous casting may be affected. The refractoriness and suitability of the sand for ferrous casting can be enhanced by adding Zircon to the sand [62]. The chemical constituents of the sands and that of the recommended mould sand in literature [36, 61, 63] were compared.

Adequacy and suitability of the moulding sands for foundry applications had been adequately discussed in detail in previous studies [45, 46]. The moulding sands' properties such as GFN, clay content, compressive strengths (wet and dry), permeability, shatter index, refractoriness, specific gravity, bulk density and flowability shown in Table 2 were found adequate and suitable (met AFS standard) for non-ferrous casting, especially aluminium casting.

The sands' physico-mechanical properties were also found suitable for metal casting since the properties favourably agreed with the AFS recommended values for casting some metals, most especially non-ferrous metals.

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Table 2. *Physico-mechanical properties of the sands against recommended/standard values*

S/N	Parameter	Average obtained Values		Recommended Standard Values	Satisfactory moulding sand properties for Aluminium casting [34]
		Belle	Oyun		
1	Moisture content	7.66%	11.31%	5-8% (for the casting of Aluminium, brass & bronze, malleable iron and medium grey iron) [60, 64].	4.5-5.5
2	Specific gravity	2.64	2.57	2.6-2.8 [65]	
3	Bulk density	1765.2 Kg/m^3	1406.25	1100 Kg/m^3 to 1800 Kg/m^3 as AFS standard for sand casting process [9].	
4	Permeability	0.10 cm/sec	0.072	0.001 cm/sec and above [65]	10-30 (permeability number)
5	Flowability	65.22%	64.29	65% [for casting of Aluminium] [64]	
6	Grain fineness number	69.01	143.02	36-90 [for non-ferrous metals [60, 64]	
7	Green compressive strength	51 kN/m^2	100.25	50-70 kN/m^2 and above [for the casting of Aluminium, brass and bronze, light grey iron and malleable iron] [60, 64]	50-70
8	Dry compressive strength	209 kN/m^2	69.73	200-500 kN/m^2 [for casting of Aluminium, brass & bronze and light grey iron] [60, 64]	200-550
9	Clay content	10%	13.42	10-12% (for the casting of Aluminium, brass & bronze, light grey iron, malleable iron, heavy steel, light steel and heavy grey steel) [60, 64]	8-10
10	Shatter index	79%	24.03	12% and above [60]	
11	Refractoriness	>900 ºC	> 1000 ºC	1100 ºC and above [60]	

The elemental compositions of the aluminium scraps are shown in Table 3. The scrap contains a high percentage of aluminium (94.85%) followed by Silicon (2.413%), Iron (0.8281%), Zinc (0.6717%), Copper (0.6226%) and Magnesium (0.38%). Other elements such as titanium, Vanadium, Manganese, Nickel, Zinc, Lead, and Zirconium are also present in small compositions.

Aluminium was found to be the main constituent of the melted scrap, with other elements such as silicon, iron, titanium, magnesium, and zinc among others in smaller proportions. Thus, the aluminium alloy belongs to the Al-Mg_2Si class of aluminium. Moreover, the presence of silicon in the alloy gives the aluminium scrap good castability. Aluminium when used for casting offers the following advantage which includes high fluidity, low melting point, low tendency for hot cracking, good castability and as-cast surface finish [63]. The presence of iron makes the aluminium hard and brittle [50].

Table 3. *Elemental chemical compositions of the aluminium scrap*

Element	**Mg**	**Al**	**Si**	**Ti**	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**
Composition (%)	0.38	94.85	2.413	0.0533	0.0175	0.0708	0.8281	0.0331	0.6226
Element	**Zn**	**Zr**	**Sn**	**Pb**					
Composition (%)	0.6717	0.0019	0.013	0.0253					

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The mechanical properties of the casts (tricycle brake pedal and motorcycle centre stand) are as presented in Tables 4-6. The mechanical properties of the casts produced have much to do with the properties of the sand used for its production [66, 67]. The properties of the aluminium casts examined include tensile, hardness and impact properties.

Tensile test was carried out on the samples of the tricycle brake pedal (TBP) and motorcycle centre stand (MCS) and the average tensile properties results are given in Figures 21-26, which include the stress at yield, stress at peak, stress at break, energy to break, elongation at yield, and young modulus of the casts.

The average stress at yield is the average yield strength exhibited by the tricycle brake pedal (TBP) and motorcycle centre stand (MCS). This is the amount of stress required to cause plastic deformation in the TBP and MCS casts and commercially available (imported) TBP and MCS. Moreover, the average stress at peak amounts to the average ultimate tensile strength (UTS) of the aluminium TBP and MCS casts and that of the commercially available (imported) TBP and MCS. It is the highest amount of stress that can be withstood before the devices' failure start to occur. The average stress at break is the stress at which the cast will totally fail. The samples of the tricycle brake pedal (TBP) and motorcycle centre stand (MCS) exhibited the same values for these stresses which are graphically presented in Figure 20 as average tensile strength.

The average tensile strength value exhibited by TBP and MCS cast is given as 124.563 and 98.795 N/mm² respectively (Figures 20). Meanwhile, the samples of commercially available (imported) TBP and MCS also exhibited an average tensile strength of 100.223 and 93.583 N/mm² respectively. The tensile strength values for both cast and commercially available (imported) TBP and MCS were comparable and the differences in their values were close, thus the casts could suitably perform well for the desired applications. The tensile strength exhibited by the TBP cast was within the minimum performance strength requirement of 65 N/mm² for a tricycle brake pedal [68]. This is an indication that the tensile properties of the brake pedal cast were suitable for its application.

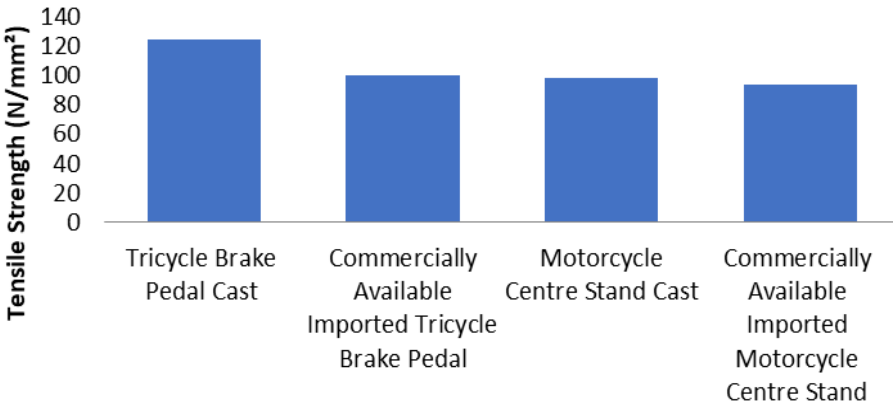

Fig. 21. *The samples of tricycle/motorcycle cast parts and commercially available (imported) parts' tensile strengths*

The energy to break is the toughness value of the cast; the amount of energy the materials can withstand or absorb before failure. The toughness value exhibited by the TBP and MCS cast is 7.870 and 11.922 Nm respectively, while the commercially available imported TBC and MCS exhibited 9.210 and 10.149 Nm respectively (Figure 22). The TBC and MCS cast has ductility values of 2.394 and 2.782 mm respectively, while commercially available imported TBC and MCS possessed 0.345 and 2.312 mm respectively (Figure 23). The strain at break is the total deformation the material will undergo before failure occurs. The value is given as 0.690% and 6.956 % for TBP and MCS cast respectively. The commercially available (imported) TBP and MCS also possessed strain at yield/break values of 5.779 and 5.985% respectively (Figure 24). The young modulus is the measure of the stiffness any of the motorcycle/tricycle cast or commercially obtained would exhibit and is given as 5789.65 and 1746.19 N/mm² for TBP and MCS cast respectively; 1776.90 and 1909.41 N/mm² for the commercially available (imported) TBP and MCS respectively (Figure 25). All these parameters are important in designing so as to determine the working conditions to which the cast products will conform. The properties exhibited by the cast products (TBP and MCS) are comparable (and even better) to that of commercially available properties.

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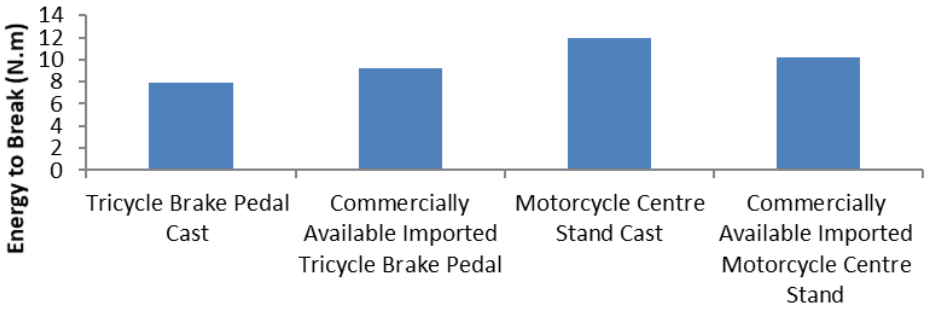

Fig. 22. *The samples of tricycle/motorcycle cast parts and commercially available (imported) parts' toughness values*

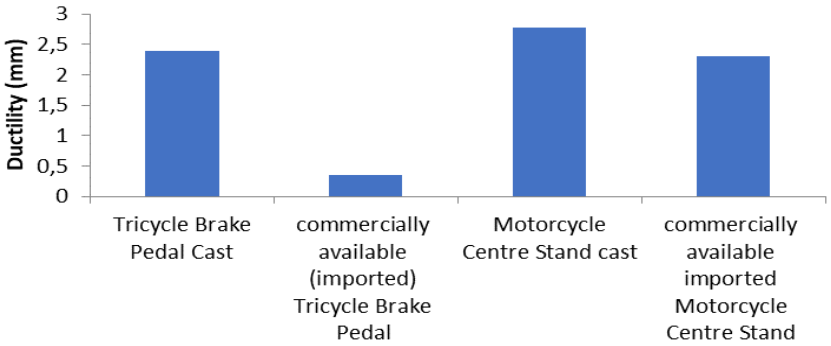

Fig. 23. *The samples of tricycle/motorcycle cast parts and commercially available (imported) parts' ductility values*

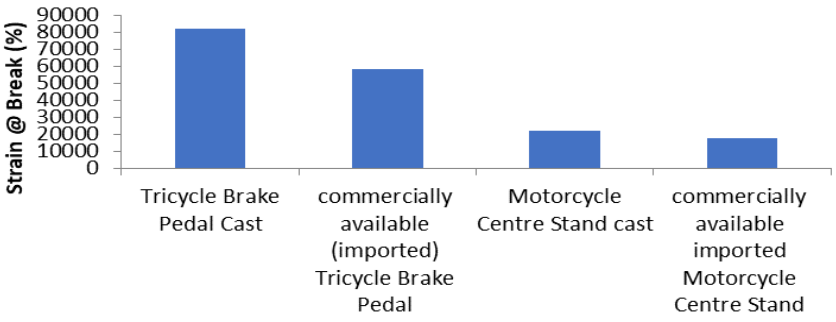

Fig. 24. *The samples of tricycle/motorcycle cast parts and commercially available (imported) parts' strain at break*

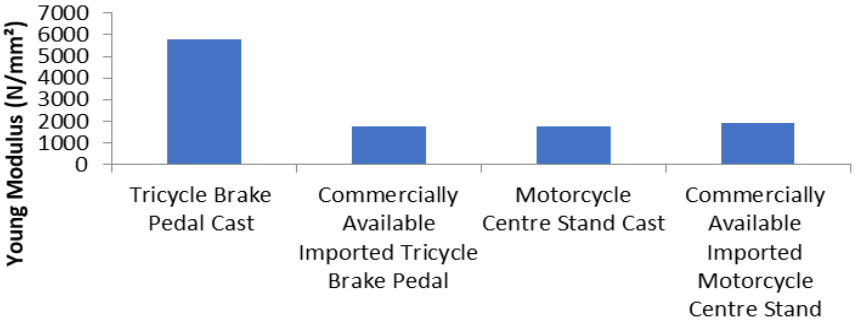

Fig. 25. *The samples of tricycle/motorcycle cast parts and commercially available (imported) parts' young moduli*

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The average (mean) hardness values for the cast products (motorcycle centre stand cast, MCP and tricycle brake pedal, TBP) and that of the imported motorcycle centre stand cast and tricycle brake pedal are presented in Figure 26. The mean hardness value exhibited by MCS cast and TBP cast was found to be 21823.00 and 81954.60 Kg/m², which are greater than the exhibited mean hardness value for MCS (21823.00 Kg/m²) and TBP (111679.000 Kg/m²).

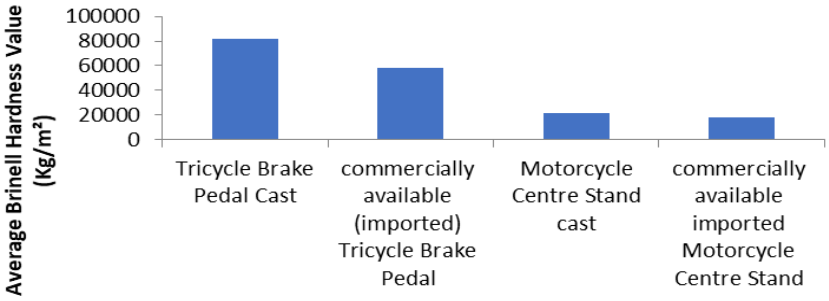

Fig. 26. *The casts and the imported brake pedal average hardness values*

The cast products may be said to be hard enough to withstand abrasiveness or deformation as a result of indentation during service, this is because of the presence of an appreciable amount of Fe in the aluminium melted scrap (as shown in Table 2). The higher the amount of Fe in aluminium the harder and brittle the aluminium will be [69, 70].

The average impact strength exhibited by the tricycle brake pedal cast and the motorcycle centre stand cast was found to be 74 J and 75 J respectively, which were the amount of sudden load each of the cast materials can withstand.

The microstructures of the tricycle brake pedal cast and the motorcycle centre stand cast at different magnifications are presented in Figures 27 and 28.

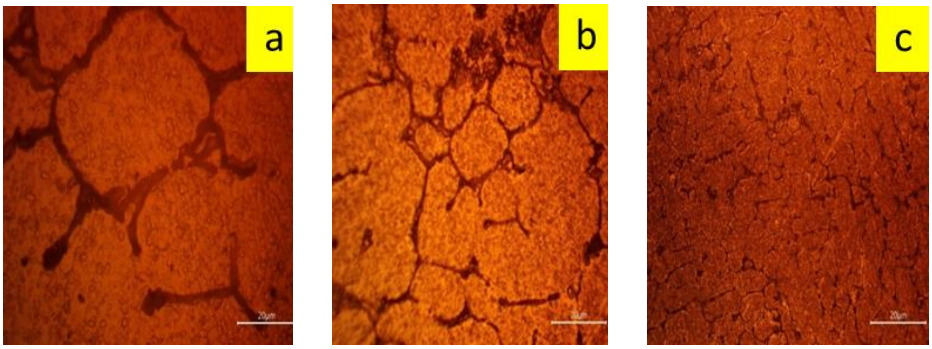

Fig. 27. *The microstructure of the tricycle brake pedal cast at different magnification: (a) x1000, (b) x400 and (c) x100*

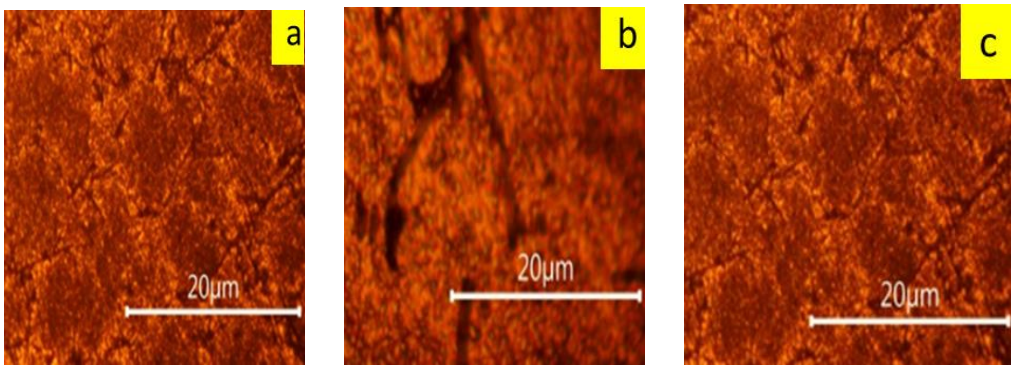

Fig. 28. *The microstructure of the cast at different magnification: (a) x1000, (b) x400 and (c) x100*

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The microstructures generally consist of fine crystals of aluminium (Al), magnesium silicide (Mg_2Si), Al-Mg_2Si phases and Al-Fe intermetallic grains. Al, Si and Mg have the highest proportion in descending order, respectively in the aluminium cast. These structures basically consist of a primary alpha solid solution of magnesium silicate in the rich solid aluminium (α) and in a matrix of eutectic magnesium silicate (Mg_2Si). Mg_2Si is the precipitate of the aluminium rich portion of Al-Mg_2Si. Mg_2Si are the dark patches in the micrographs. The casts aluminium belongs to the Al-Mg_2Si class of aluminium alloy as revealed in the chemical compositions results of the aluminium alloy in Table 2.

4. Conclusions

The following conclusions were drawn from the study:

i. Belle and Oyun natural moulding sands were found suitable for casting and production of motorcycle centre stand and tricycle brake pedal (respectively) of acceptable standards using aluminium scraps. The cast products exhibited an adequate tensile strength of 122.563 and 98.795 N/mm² by tricycle brake pedal and motorcycle centre stand sample respectively; and average Brinell hardness value of 21823.00 and 81954.60 Kg/m² by MCS cast and TBP cast respectively, which were greater than the exhibited mean hardness value of 21823.00 and 111679.000 Kg/m² exhibited by commercially available (imported) Motorcycle cycle stand and Tricycle brake pad respectively.

ii. The aluminium tricycle brake pedal and motorcycle centre stand produced through sand casting processes (using locally available moulding sands) exhibited adequate properties like strengths and hardness (wear resistance) to withstand their applications and were found comparable with the commercially available exported ones. Thus, the production of such motorcycle/tricycle and related components can be domesticated in Nigeria, which would enhance the sustainability of small and medium scale enterprises required for job creation, enhance the nation's self-reliance through the reduction in the importation of goods and boost Nigeria's economic status.

iii. The effective utilization of wastes like aluminium scrap in the production of transportation devices components (such as tricycle brake pedal and motorcycle centre stand) will greatly assist Nigeria, as a country, in achieving perfect sustainable waste management and safe health environment. This is due to the fact that the transformation of the raw materials into finished products using locally available raw materials in the development of technology is a significant factor towards successful industrialization.

iv. The consequences of the Covid-19 pandemic, compounded by the over-reliance on the oil and gas industry coupled with excessive proliferation of foreign commodities to satisfy citizenry needs and lack of commitment to other economic sectors, such as insufficient natural resource exploitation and low industrialization contributed to the Nigeria economic recession with negative growth for five quarters. This can be positively addressed by developing Nigeria's foundry technology sector through effective utilization of available natural moulding sands (such as Belle and Oyun moulding sands) for the production of motorcycle/tricycle components.

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PERCEIVED CHARACTERISTICS OF MATERIALS IN A PRODUCT AESTHETICS CONTEXT

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ABSTRACT

Materials possess a number of objective characteristics, the values of which can be determined experimentally. On the other hand, materials also have a number of subjective characteristics, such as perceived quality, perceived performance, and so on. These perceived features are very important in constructing the product's aesthetics. The paper presents the results of three experiments performed to determine perceived characteristics (quality, performance, durability, modernity, and aesthetics) associated with four classes of materials (metal, plastic, wood, and ceramics). Computer-designed product models were used in the experiments. All products used in the experiment were low-tech. The design was varied on three levels: minimal, elaborate, and exceptional. Differences in perception were found for each level of design. It has been found that the elaborate design improves the perception of the characteristics of the materials, but the exceptional design has a negative influence on the perception. Plastics benefit greatly from the contribution of design, while wood (traditional material) is better perceived in the case of minimal design.

KEYWORDS: materials perception, product aesthetics, perceived quality, perceived performance, perceived durability

1. Introduction

Materials have an important contribution to product aesthetics, given that each material is perceived differently through the senses. Therefore, the choice of materials is an important task for the product designer.

The process of selecting materials is very complex, depending on a number of characteristics of different nature: functional, technological, financial, aesthetic, maintenance, etc. Although these characteristics are often considered separately (which is not correct), their action is synergic. A competent designer should consider and balance these different characteristics in the selection of materials to ensure that the product will not only fulfil its functions but will also have a proper aesthetic appearance, leading to a positive experience for the user.

Today, the designer has at her/his disposal numerous informational resources for the selection of materials, such as textbooks, manuals, databases, the internet, computer applications, examples of good practices, etc. [1]. However, the range of materials available to the designer is very large and constantly growing. It is estimated that there are over 100,000 materials [2]. The selection of materials is increasingly complex and has become a critical component of the design process [3].

The fact that the technical characteristics are easier to determine does not mean that they are more important than the aesthetic characteristics. It does not make sense to define technical characteristics in great detail and ignore other characteristics [3]. Moreover, Gant [4] indicated that materials are vectors through which designers create deep emotional connections between products and their contemplators.

Each material has distinctive characteristics that the designer can exploit as a source of inventiveness, while at the same time other characteristics are constraints. It is a well-known fact that new materials allow and even stimulate designers to create new shapes, colours, and textures and to involve users in new ways of using products. "Form Follows Materials" is the paraphrase of Michael Ashby [5] which emphasizes the strong influence of materials in shaping products.

Products material greatly influences the range of functions, durability, associated costs, perceived

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quality, user feedback, and user experience. When users interact with products, their senses come into contact with the materials from which these products are made, especially their casings. Users notice the colours of the materials, feel their texture, local deformation, and weight, and hear the sounds that the materials produce when the product is working [6].

Designers use materials as vectors to generate complex sensory experiences when people come in contact with products. In these cases, the materials are used as codes that convey meanings for generating emotions. For example, wood and ceramics are frequently used in Zen-inspired design, considered as a balance between general and detail, simplicity and ease of use [7].

By reducing the complexity of the material selection process to the extreme, the designer is put in a position to take into account the practical needs, but also the meanings that the materials convey. The designer asks questions such as: "Due to the material chosen, will the product express quality, ease of use, convenience, durability?" [8]. It would be beneficial for designers to have a dictionary of meanings for different materials.

An important axiological aspect of the material's meaning is that people use materials in interiors created or designed by themselves to build a strong personal identity. So, the materials convey the cardinal values of the owner [9].

What is known today about the meanings of materials is the result of the process of recording society's general impression, records made by theorists of the field in a relatively subjective manner. Objective approaches based on scientific experiments are rare.

Thus, ceramic is associated with stiffness, coldness, and weight (especially due to its use for festive dinner services) [10]. Ceramic is perceived as a hygienic and abrasion-resistant material. Metals offer strength (perceived, but also real), toughness, but are felt like a cold and distant materials, like most artificial materials. Following the Industrial Revolution, metals became associated with high precision, technological superiority, and economic power [11]. Wood is perhaps the warmest and closest to the human spirit of all existing classes of materials. It is easy to process, and its anisotropy is often used as inspiration by creators. It has a strong association with the notion of craftsmanship.

The most debated class of materials in terms of meanings is indisputably that of plastics. Manzini [12] even argued that there was a "loss of meaning" in the world of materials because of plastic. At the beginning of the twentieth century, plastics were considered symbols of progress and modernism. They successfully replaced traditional materials such as metal, wood, and ceramics. Interwar designers made new shapes with the help of thermosetting materials (bakelite, melamine, etc.), then plastics went through a period of adoration in the sixties of the last century to fall into the position of hated materials, being associated with kitsch, cheapness and, especially pollution.

Katz [13], but also most specialists, justifiably believed that plastic products have organic forms due to a technological condition, namely the requirement for the semi-liquid material to flow into the injection mould. This is the cause of the appearance of so many elongated and rounded products in the creation of designers such as Eero Aarnio, Verner Panton, Joe Colombo, Gino Colombini, etc. Technological conditions obviously also influence the shapes of products made from other classes of materials.

The semantic correlation between materials and forms was also investigated. It has been found that people associate certain materials with certain shapes as a result of their daily experience of using products in which the casing from a certain material has a certain shape [14]. For example, metals are presented in products with flat surfaces and straight edges, and plastics in products with rounded shapes. In fact, technological requirements have imposed these correlations.

Another direction of research was to determine the tactile perception of quality and performance associated with different materials. For example, Dumitrescu [15] studied the correlation between a series of parameters (quality, performance, price, warmth, and aesthetic preference) tactile perception, and different classes of materials, focusing on the class of wood.

From the study of the dedicated literature, it can be observed that there is little research aimed at the correlation between certain classes of materials and the perception of certain characteristics of the products. Such research would be useful to draft a designer's guide to the choice of materials, so that the product would gain certain perceived characteristics such as quality, durability, etc.

2. Method

Following the study and assimilation of the specialised literature, the following research objectives (RO) have emerged:

RO1. Establishment of a hierarchy of the main classes of materials according to a series of significant perceived characteristics.

RO2. Study the influence of design on the perception of the considered characteristics according to the generic classes of materials. The levels of parameter design to be considered were: minimal, elaborate, and exceptional.

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RO3. Study the influence of the mode of display on the perception of the considered characteristics.

For RO2, a number of null hypotheses were also formulated. In order not to take up too much space with repetitive statements, only the generic format of the working hypotheses is indicated below:

H0XY: The perception of the characteristic X for a product made of material Y is the same regardless of the type of design (minimal, elaborate, or exceptional).

In order to perform the experiment, the following classes of materials were chosen:

- wood;
- metal;
- plastic;
- ceramics.

And the following perceived characteristics were taken into account:

- quality;
- performance;
- durability;
- modernity;
- aesthetics.

To avoid the bias of results, it was decided that the products used in the experiment should not be computer, electronic or other high-tech types. Finally, the following product subclasses were chosen: desk lamp; stool; coat hanger; citrus juicer; soap dish; ashtray and sticky note compartment. It was also decided that the structural and functional complexity of the products should be at a reduced level, so the materials would be more important to the observer. In order to obtain the images to be used in the experiment, the products were modelled in Catia software by a student enrolled in a master's programme in computer-aided design. The distinction between the different classes of materials was achieved in terms of colour and texture.

Fig. 1. *Wooden stool – elaborate design*

Three levels of design were chosen: minimal, elaborate, and exceptional. The minimal design referred to the products with a simple and functional look. The elaborate design included products that show a certain refinement of shapes, colours, and textures to improve the appearance. The exceptional design referred to products in which the designer's intervention was radical and changed to some extent the archetypal structure of the product. For products with exceptional design, the sources of inspiration were the creations of famous designers such as Philippe Starck or Stefano Giovannoni. Figures 1-4 show examples of product images.

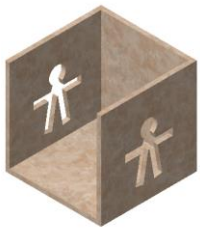

Fig. 2. *Ceramic sticky notes compartment-exceptional design*

Fig. 3. *Plastic citrus juicer-exceptional design*

Fig. 4. *Metallic soap dish-minimal design*

It was decided that the participants in the experiment would evaluate the products using an electronic questionnaire in which each product image would be followed by the following instruction: "Please assess the above product against the following characteristics:", followed by the list of five characteristics. The assessment would be performed using 5-point Likert scales.

In order to avoid the fatigue and boredom of the participants, three separate experiments were organised. In Experiment 1, the design levels were minimal and elaborate, and the products used were: a lamp, a stool, and a coat hanger. In Experiment 2, the design levels were minimal and exceptional, and the products used were: a citrus juicer, a soap dish, and an ashtray. Experiment 3 targeted Research Objective 3, and the design levels were minimal and

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exceptional, and the product used was a sticky note compartment. In this experiment, in the first stage, the products were shown in separate images, and in the second stage, the four products corresponding to the four materials were presented in a single image.

3. Results

3.1. Experiment 1

The experiment was carried out with 203 participants (121 women and 82 men). All participants were students enrolled at a large technical university in Romania. The participants had basic training in product aesthetics. The accuracy of the results was tested using Z-score. No Z-scores were outside the interval [-3; +3], so no data sets were eliminated. The Z-score ranged between -2.81 and 2.50. The reliability of data was tested using Cronbach's alpha coefficient. The calculated value for the whole set of data was $\alpha = 0.972$, a value which stands for very good reliability.

After collecting all the results, the average values were calculated for each material against each perceived characteristic. The results are displayed in Tables 1-4.

Table 1. *Overall mean values for perceived characteristics in Experiment 1*

	Quality	Performance	Durability	Modernity	Aesthetics
Plastic	3.058	3.034	3.162	3.024	2.807
Metal	3.294	3.132	3.470	3.180	2.999
Wood	3.019	2.943	3.126	2.834	2.703
Ceramic	2.984	2.993	3.245	2.904	2.742

Table 2. *Mean values for perceived characteristics for minimal design in Experiment 1*

	Quality	Performance	Durability	Modernity	Aesthetics
Plastic	2.926	2.898	3.079	2.646	2.477
Metal	3.200	3.102	3.391	2.957	2.931
Wood	3.020	2.959	3.158	2.620	2.721
Ceramic	2.734	2.885	3.085	2.556	2.511

Table 3. *Mean values for perceived characteristics for elaborate design in Experiment 1*

	Quality	Performance	Durability	Modernity	Aesthetics
Plastic	3.190	3.169	3.245	3.402	3.136
Metal	3.388	3.163	3.548	3.402	3.067
Wood	3.018	2.928	3.095	3.048	2.685
Ceramic	3.233	3.102	3.406	3.253	2.974

Table 4. *Difference (elaborate – minimal design) between mean values in Experiment 1*

	Quality	Performance	Durability	Modernity	Aesthetics
Plastic	0.264	0.271	0.166	0.756	0.659
Metal	0.187	0.061	0.158	0.445	0.136
Wood	-0.002	-0.031	-0.062	0.427	-0.036
Ceramic	0.499	0.217	0.320	0.697	0.463

When overall means (Table 1) were considered, the ranking of materials was clear and also uniform for the five characteristics considered: quality, performance, durability, modernity, and aesthetics. The hierarchy was: metal, plastic, ceramic, and wood, with the observation, that the differences were very small (negligible) between the last two materials in the case of quality and performance.

In the case of minimal design (Table 2), there was a change in the hierarchy, respectively the plastic was overtaken by wood and sometimes even ceramics. Even if the differences were small, the change in the hierarchy was significant. When the design did not affect the perception, the hierarchy corresponded to the reality, respectively the wood had superior perceived quality and durability compared to

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plastic. Plastic retained the advantage of perceived modernity, especially since it is a relatively recent material.

When the influence of elaborate design intervened (Table 3), the plastic was favoured especially in terms of aesthetics. It should be noted that design significantly improved the perception of the plastic characteristics.

After making the differences between the mean values of the product characteristics with an elaborate design and those corresponding to minimal design (Table 4), it was found that the elaborate design positively influenced the perception of all characteristics of materials, with one notable exception: wood. So, the elaborate design was unfavourable to wood products, but modernity had to gain; it should not be forgotten that wood is the oldest material used by man and is considered to be the paradigm of traditional material.

Table 5. *Results of T-Test - Paired Two Sample for Means (minimal design-elaborate design)*

***t*(202) = 1.65**		***p* < 0.05**	***Difference is***
Variation of quality for wooden products	-2.71	0.0037	significant
Variation of quality for metallic products	-3.01	0.0014	significant
Variation of quality for plastic products	-4.47	6.3×10^{-6}	significant
Variation of quality for ceramic products	-6,53	2.5×10^{-10}	significant
Variation of performance for wooden products	-1.37	0.08	not significant
Variation of performance for metallic products	-1.48	0.069	not significant
Variation of performance for plastic products	-2.57	0.059	not significant
Variation of performance for ceramic products	-4.66	2.81×10^{-6}	significant
Variation of durability for wooden products	-1.59	0.0056	significant
Variation of durability for metallic products	-2.06	0.021	significant
Variation of durability for plastic products	-3.83	8.31×10^{-5}	significant
Variation of durability for ceramic products	-6.33	7.73×10^{-10}	significant
Variation of modernity for wooden products	-8.65	7.9×10^{-16}	significant
Variation of modernity for metallic products	-7.95	6.18×10^{-14}	significant
Variation of modernity for plastic products	-9.67	9.6×10^{-19}	significant
Variation of modernity for ceramic products	-13.8	1.67×10^{-31}	significant
Variation of aesthetics for wooden products	-3.17	0.0008	significant
Variation of aesthetics for metallic products	-1.59	0.0056	significant
Variation of aesthetics for plastic products	-5.56	4.13×10^{-8}	significant
Variation of aesthetics for ceramic products	-9.29	1.21×10^{-17}	significant

The question was whether there was a true difference between the perception of the product characteristics with a minimal design and those with an elaborate design. It should be kept in mind that 5-point Likert scales were used, and a difference of 0.1 was actually just 2.5%. In such a situation, it is recommended to use a t-test - paired two samples for means. The generic null hypothesis was: H0: The perception of the characteristic X for a product made of material Y is the same regardless of the type of design (minimal or elaborate). (The corollary of this hypothesis was that the difference was insignificant.) In order to reject the hypothesis (and the difference to be significant), the p-value should be < 0.05. Table 5 contains the results of the application of the t-test - paired two samples for means. The difference was significant in the vast majority of cases except for the performance characteristics of wood, metal, and plastic. In other words, the perceived performance of these materials was not influenced by design. People did not expect these materials to work better if people saw them in a higher aesthetic context.

3.2. Experiment 2

Experiment 2 was carried out with 152 participants (93 women and 59 men). All participants were students enrolled at a large technical university in Romania. The participants had basic training in product aesthetics. No participants were involved in Experiment 1. The accuracy of the results was tested using Z-score. No Z-scores were outside the interval [-3; +3], so no data sets were eliminated. The Z-score ranged between -2.18 and 2.51. The reliability of data was tested using Cronbach's alpha coefficient. The calculated value for the whole set of data was α = 0.98, a value which stands for very good reliability.

After collecting all the results, the average values were calculated for each material against each perceived characteristic. The results are displayed in Tables 6-7. Not all results are presented because some are very similar to those results obtained in Experiment 1.

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Table 6. *Mean values for perceived characteristics for exceptional design in Experiment 2*

	Quality	**Performance**	**Durability**	**Modernity**	**Aesthetics**
Plastic	2.796	2.776	2.978	2.798	2.526
Metal	2.925	2.857	3.160	2.943	2.643
Wood	2.748	2.708	2.919	2.781	2.412
Ceramic	2.932	2.846	3.107	2.897	2.660

Table 7. *Difference (exceptional – minimal design) between mean values in Experiment 2*

	Quality	**Performance**	**Durability**	**Modernity**	**Aesthetics**
Plastic	-0.331	-0.340	-0.235	-0.221	-0.390
Metal	-0.373	-0.355	-0.366	-0.151	-0.384
Wood	-0.274	-0.300	-0.401	-0.072	-0.406
Ceramic	-0.428	-0.421	-0.366	-0.344	-0.577

Table 8. *Results of T-Test - Paired Two Sample for Means (minimal design-exceptional design)*

	***t*(151) = 1.65**	***p* < 0.05**	***Difference is***
Variation of quality for wooden products	4.77	2.15×10^{-6}	significant
Variation of quality for metallic products	6.54	4.33×10^{-10}	significant
Variation of quality for plastic products	5.58	5.18×10^{-8}	significant
Variation of quality for ceramic products	8.16	5.95×10^{-14}	significant
Variation of performance for wooden products	5.42	1.12×10^{-7}	significant
Variation of performance for metallic products	6.15	3.31×10^{-9}	significant
Variation of performance for plastic products	6.25	1.99×10^{-9}	significant
Variation of performance for ceramic products	7.29	8.17×10^{-12}	significant
Variation of durability for wooden products	4.67	3.31×10^{-6}	significant
Variation of durability for metallic products	6.42	8.21×10^{-6}	significant
Variation of durability for plastic products	4.08	3.65×10^{-5}	significant
Variation of durability for ceramic products	6.12	3.83×10^{-9}	significant
Variation of modernity for wooden products	1.05	0.14	not significant
Variation of modernity for metallic products	2.15	0.016	significant
Variation of modernity for plastic products	3.4	0.0004	significant
Variation of modernity for ceramic products	4.96	9.13×10^{-7}	significant
Variation of aesthetics for wooden products	5.38	1.38×10^{-7}	significant
Variation of aesthetics for metallic products	7.5	2.47×10^{-12}	significant
Variation of aesthetics for plastic products	5.82	1.66×10^{-8}	significant
Variation of aesthetics for ceramic products	7.68	9.09×10^{-13}	significant

There were certainly many similarities in the hierarchy of materials obtained in the two experiments. The most remarkable difference was the rise of ceramics in the case of exceptional design, which ended up achieving values very close to the metal or even exceeding it (Table 6).

Surprising were the differences between the means of characteristics; the perception of products with an exceptional design was lower than those associated with products with a minimal design (Table 7). The situation was the same for all characteristics and all materials. It can even be said that the exceptional design acted against the perception of characteristics. A direct conclusion was that when a manufacturing company would rely on the good reputation of the materials used, then the design should be elaborate at most.

As in the case of Experiment 1, the question of the authenticity of the observed differences was raised. The t-test - paired two samples for means was applied again and the null hypothesis was: H0: The perception of the characteristic X for a product made of material Y is the same regardless of the type of design (minimal or exceptional). In order to reject the hypothesis (and the difference to be significant), the p-value < 0.05. Table 8 contains the results of the application of the t-test - paired two samples for means. The difference was significant in the vast

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majority of cases with only one exception: the modernity of wood products. This was expected because the wood was perceived as a traditional/classic material and no aesthetic trick can change that.

3.3. Experiment 3

The purpose of Experiment 3 was not to reconfirm the results obtained in the first two experiments but to investigate possible differences generated by the mode of display. Is influenced the perception of material characteristics if the products are displayed separately or together?

Experiment 3 was carried out with 211 participants (114 women and 97 men). All participants were students enrolled at a large technical university in Romania. The participants had basic training in product aesthetics. No participants were involved in the previous experiments. The participants assessed the products (sticky notes compartments) presented one per image and after one week the same participants assessed the same products but displayed four on one image (corresponding to the four materials). The accuracy of the results was tested using Z-score. No Z-scores were outside the interval [-3; +3], so no data sets were eliminated. The Z-score ranged between -1.95 and 1.25. The reliability of data was tested using Cronbach's alpha coefficient. The calculated value for the whole set of data was $\alpha = 0.958$, a value which stands for very good reliability.

Table 9. *Difference (one product per image against all products per image) between mean values*

	Quality	Performance	Durability	Modernity	Aesthetics
Plastic	-0.100	-0.175	-0.100	-0.152	-0.197
Metal	-0.187	-0.197	-0.045	-0.204	-0.299
Wood	-0.088	-0.211	-0.062	-0.190	-0.178
Ceramic	-0.185	-0.211	-0.081	-0.218	-0.415

The differences between mean values are displayed in Table 9. It can be easily observed that in all cases the presentation of all four products together led to a positive assessment. So, in general, it is better to display the products together to improve the quality of assessment.

4. Discussion and Conclusions

From the very beginning, it should be noted that the experiments were well designed and that the participants were positively involved in the run of experiments. No Z-Score data were removed, and Cronbach-alpha values were very high (> 0.958) in all cases.

In the three experiments, there were considered five material characteristics with an important role in public perception: quality, performance, durability, modernity, and aesthetics. Given that the assessment was made from a design perspective, purely technical characteristics such as yield strength, electrical conductivity, etc. were not taken into account, as they were insignificant to the general public.

The generic materials subjected to experiments were chosen from the same perspective of relevance to the general public. Thus, no polymer matrix composite materials were selected, because it would be difficult for the public to distinguish them from ordinary plastics. Glass had also been ignored because it had similar properties to ceramics, except for transparency.

The design was varied on three levels: minimal, elaborate, and exceptional. The minimal level corresponded to products with a simple and functional appearance. The elaborate level corresponded to products that display a certain refinement of shapes, colours, and textures to improve the appearance. Exceptional level corresponded to products in which the designer's action was radical and altered to some extent the archetypal structure of the product.

After the analysis of the results of Experiment 1, a partially unexpected perception hierarchy of materials emerged: 1 - metal; 2 - plastic; 3-4 - wood and ceramics. The surprise was the plastic, which surpassed materials with superior values of intrinsic characteristics. The explanation was that the elaborate design favours plastic.

When the design was at the minimal level, the hierarchy of materials was the natural one, with wood surpassing plastic in the relevant characteristics. But when the level of design was elaborated, all the characteristics of the materials were perceived with superior values for metal, plastic and ceramics. Moreover, plastic, and ceramics values have made a substantial increase in modernity and aesthetics. The situation was remarkable for ceramics, considering that was a traditional material. However, another traditional material, wood, marked setbacks in the case of elaborate design. It was obvious that the connotation of wood as traditional material did not match the upscaling given by the design.

When the level of design varied from minimal to exceptional (Experiment 2), a remarkable situation was observed, namely all values for all materials and all characteristics associated with the exceptional design were lower than those associated with minimal design. There was only one conclusion: the excessive design was detrimental to the perception of material characteristics. The excessive design brought certain advantages, but it certainly did not improve the perception of materials.

Regarding the presentation of the same product in different options of material (Experiment 3), it was found that the presentation in the same image of the options contributes positively to their evaluation.

The previous comments regarding the differences in perception introduced by the different levels of design were true, being supported in the vast majority of cases by the results obtained after the application of the t-tests.

5. Limitation and Future Research

One limitation was given by the segment of participants: students at a technical university. They were people who have studied disciplines such as: materials, the strength of materials, etc. They knew well the characteristics such as yield strength, resilience, corrosion resistance, etc. This influenced their perception of materials. It could be said that it was an anti-bias influence because they knew the reality well and they were not biased by the levels of design or the possible connotative aspects of the different classes of products.

Another limitation was given by the type of products used in the experiment, respectively low-tech products. Only one product was electric. No electronic or IT products were used. The structural and functional complexity of the products has been at a low level.

As not only the considered five material characteristics (quality, performance, durability, modernity, and aesthetics) are important for the observer, the subsequent research will and should focus on new perceptual characteristics such as elegance, prestige, finish, structural integrity, etc.

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STUDIES ON EPOXY RESIN MODIFIED WITH ORGANIC AGENTS

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ABSTRACT

Epoxy composites are one of the most versatile thermosetting polymers widely used due to their strong soldering resistance, high insulation, and thermal resistance. A wide range of raw materials can be used for the preparation of epoxy resins, thus offering a variety of resins with highly controllable performance characteristics. Essentially, epoxy resins are low molecular weight liquids with two or more epoxide functional groups. To get superior properties of the composite material, a broad variety of inorganic and organic modification agents are used. For this study, two protein substances, gelatin, and wheat gluten were used to change the basic properties of three types of epoxy resins. The specific heat of modified epoxy resin materials was performed by using Differential Scanning Calorimetry (DSC) instrument. The thermal analysis was developed on two heating-cooling cycles and the specific heat was evaluated for each segment of the cycle analysis. Also, the mechanical behavior of organic modified polymers subjected to compression was studied using INSTRON compressive testing machine.

KEYWORDS: epoxy resins, organic compounds, thermal properties

1. Introduction

Epoxy resins as important organic matrices, thanks to their chemical structure and the possibility of modification, have unique properties, which contribute to the fact that these materials have been used in many composite industries for many years. [1]. Epoxy resins are repeatedly used in exacting applications due to their exquisite mechanical properties, thermal stability, scratch resistance, and chemical resistance. Moreover, epoxy materials also have really strong resistance to solvents, chemical attacks, and climatic aging [2-3]. The presented features confirm the fact that there is a constant interest of scientists in the modification of resins and understanding their mechanisms, as well as in the development of these materials to obtain systems with the required properties.

Protein polymers are natural macromolecules derived from plants and animals which makes them an easily obtainable, renewable resource. Therefore, the development of environmentally friendly epoxy systems is of great importance for designing green and biocompatible materials in many applications.

Wheat gluten is a plant protein that is unique among plant proteins, because of its availability, good biodegradability, low price, unique viscoelastic properties, and ability to crosslink on heating [4, 5]. Gelatin is a natural mixture of polypeptides that is obtained from collagen Type I. Collagen used for gelatin extraction that can be isolated from bones, tendons, and skin of animals, but it is especially obtained from bovine and porcine [6]. Also, gelatin is a low-cost and abundantly available protein biopolymer obtained from partial hydrolysis of collagen found in various biomaterials [7]. It has been widely explored in biomedical applications such as tissue engineering, drug delivery, and wound healing [8]. The cross-linked gelatin nanofibers showed environmental stability, better mechanical properties, and comparable filtration performance [9].

Many researchers had investigated the mechanical, chemical, and thermo-mechanical properties of different epoxy resins systems by different methods [10-12].

Thermal analysis with DSC 1 Mettler Toledo (Differential Scanning Calorimetry) is used to calculate the degree of crystallinity and its composites, but account must be taken for melting and recrystallization that simultaneously occur when heating [13, 14].

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The specific heat of a certain material is a physical quantity that is characterized by the energy exchange between the respective material and the environment, energy exchange that has the effect of changing the temperature value of the material. To determine the specific heat of the polymeric materials, the heat flux signal emitted by the specimen to be analyzed is compared with the known signal of the reference specimen.

Application of the DSC test method to polymer matrix composites may also encounter difficulties due to specimen mass loss from either moisture evolution or material decomposition, but this problem can be overcome by taking proper precautions [15-17].

The main purpose of this study was to analyse the composite materials modified with organic agents (wheat gluten and gelatin) with a matrix made from three different epoxy resins, in particular specific heat. There is a multitude of mechanical tests, and because the stresses to be transposed are diverse, they can be performed in static or dynamic conditions, at ambient temperature, as well as at high or low temperatures, in air or in corrosive environments, etc. The determination of the mechanical properties of compression of the composite materials modified proteins was performed as experiments, on the mechanical testing machine INSTRON 8030.

2. Materials and methods

The study was carried out using three epoxy resins, namely Epiphen RE4020-DE 4020, Epoxy Resin C, and Epoxy Resin HT2 [18-20]. Wheat gluten and gelatin were purchased from Sigma-Aldrich and were used as received. The preparation of the modified composites consists of the addition of a quantity of modifying agents in the form of wheat gluten powder and gelatin to the epoxy matrix.

The first step in the process of forming protein-modified composites is to establish the preparation protocols of the moulds (cylindrical polypropylene tubes). These types of moulds were chosen precisely so that the materials could be easily extracted after the period of polymerization (hardening), without being damaged. The modification of the epoxy system consisted of the introduction of a quantity of modifying agents (gluten and gelatin) in the proportion of 1%, 2%, and 3% weight ratios. The resulting mixture was mixed mechanically (500 rpm) for 24 hours at 60 degrees Celsius.

The next step in the process of forming the materials was the actual pouring of the mixture into moulds. Twenty-four hours later, the materials were extracted from the polypropylene tubes. In the final step, the polymerized materials are treated in the heat treatment furnace. The parameters after which the heat treatment was carried out were established by working protocols, namely: heating the enclosure to a temperature of 60 degrees Celsius, maintaining the time for 8 hours, raising the temperature to 80 degrees Celsius, maintaining it for 2 hours, and finally, the last stage consisted of heating to a temperature of 90 degrees Celsius for 1 hour. The purpose of applying the heat treatment is to eliminate the residual stresses that can appear in the material during casting or during the polymerization period, giving it a necessary state of equilibrium. After the materials had cooled, specimens were extracted (according to the standards in force), in order to determine the parameters that will characterize the composite materials formed depending on the type and quantity of the modifying agent used.

The thermal measurements were performed by using Differential Scanning Calorimeter (DSC) for specific heat analysis. This device is equipped with a sensor that checks the atmosphere inside the cell and provides a balance between the sample to be analyzed and the reference sample. The specific heat was determined from the heat absorption curve and/or the released heat for each type of material. The specific heat of epoxy composite materials was investigated by heating the samples at temperature intervals of -60 to 150 °C and by cooling at temperature intervals of 150 to -60 °C, with 10 °C/min, and the cycle is repeated. Both heating and cooling DSC curves can provide information on the nature of samples.

The behavior of the composite material to the mechanical stresses produced by external forces depends on certain specific properties of it, called mechanical properties. Most tests involve the application of test tubes until the material yields and the strength limit is reached. Also, the mechanical tests were performed on an Instron testing machine and the conditions for testing were established at a speed of 55 mm/min and the stop condition was set at a 40% drop of the loading force. The applied load was 2 kN for bending and 25 kN for the compression test. Samples extraction for the mechanical analysis was performed after the thermal treatment. They have been of 160 mm in length ensuring a length of 10 mm as an engagement zone.

3. Results and discussion

In order to determine the specific heat of the polymeric materials, the heat flux signal emitted by the specimen to be analyzed is compared with the known signal of the reference specimen. This process consists in recording an isothermal curve at low temperature, after which, as the temperature increases, another isotherm is recorded. The specific heat is determined by the quantitative measurement of the energy absorbed by the material, depending on the temperature.

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Analyzing the data in the diagram of the unchanged C resin and the modified one with an organic modifying agent, we noticed that after the first heating there is a keeping of the test specimens, which means the removal of organic modifiers from the epoxy resin. Thermal analysis was performed at different temperature ranges, starting from -50, 50 ºC, values that are shown in the diagrams.

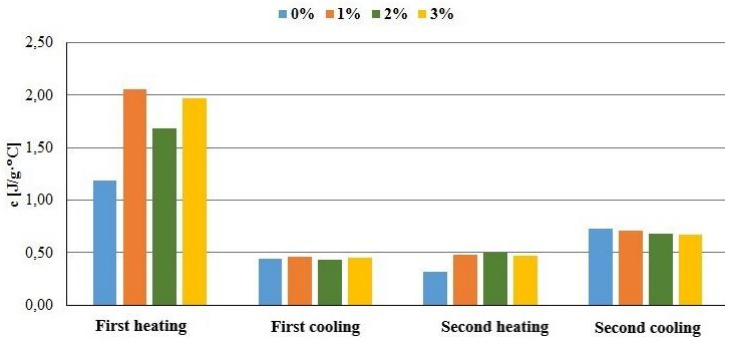

Fig. 1. *Specific heat values of C modified epoxy resin materials (-50, 50 ºC temperature interval)*

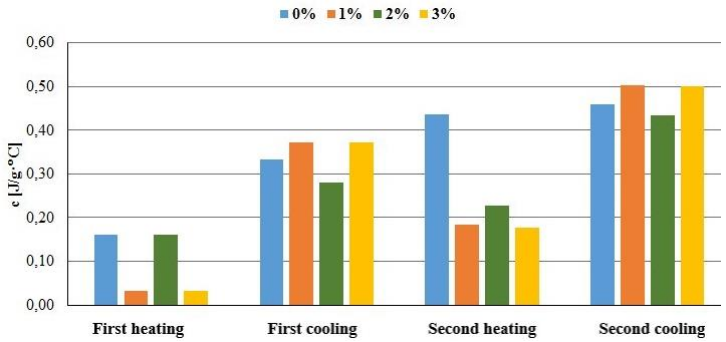

Fig. 2. *Specific heat values of E modified epoxy resin materials (-50, 50 ºC temperature interval)*

In the case of materials formed with epoxy resin E, a considerable change is observed at the second cooling, in which all the resins are almost identical, which means that the modifiers give the material high thermal properties.

In Figure 3, we observed an almost identical behavior of the material at the first heat of the modified material with gelatin and gluten, as well as the unmodified one, as a result of which at the first cooling there are improved values compared to the control specimen.

According to the type C epoxy resin diagram at 90, 140 ºC, the value of the specific heat at the first heating, still shows a significant increase, especially the control C0 and C2, which has very good property compared to the epoxy matrix materials of the same category. The highest specific heat values were obtained for modified and unmodified type C epoxy composites.

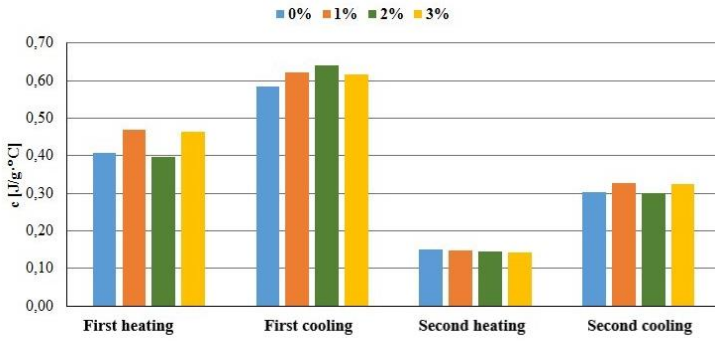

Fig. 3. *Specific heat values of HT modified epoxy resin materials (-50, 50 ºC temperature interval)*

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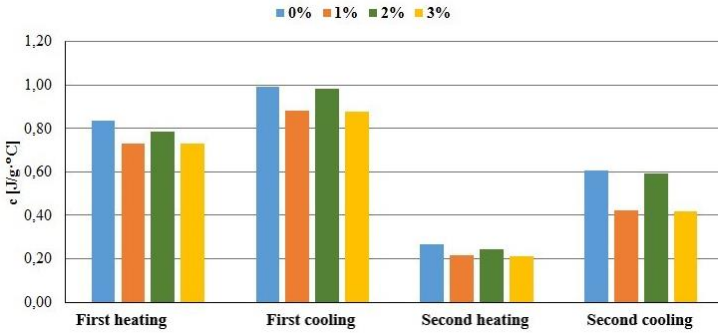

Fig. 4. *Specific heat values of C modified epoxy resin materials (90, 140 ºC temperature interval)*

In the case of materials with epoxy matrix type E, it is observed that combustion occurs in materials E0 and E2 at the first heating.

Thus, higher values of specific heat for E3 material were obtained. No significant variations in the specific heat values of the type E epoxy materials were observed during cooling.

HT type epoxy matrix composite materials showed variations in specific heat values depending on the type of modifying agent used in the structure of the studied materials. We observed higher values of the resin after the first heating showing high thermal stability, where HT0 and HT2 have similar values, as well as HT1 and HT3.

Before and after determining the specific heat values of the materials, the samples were weighed to determine the loss of substance caused by the increase in temperature. This loss is due to: polymer wastewater, small amounts of diluent or other volatile compounds, which are in the mass of the polymer in the form of oxygen and carbon dioxide resulting from chemical reactions, being represented graphically.

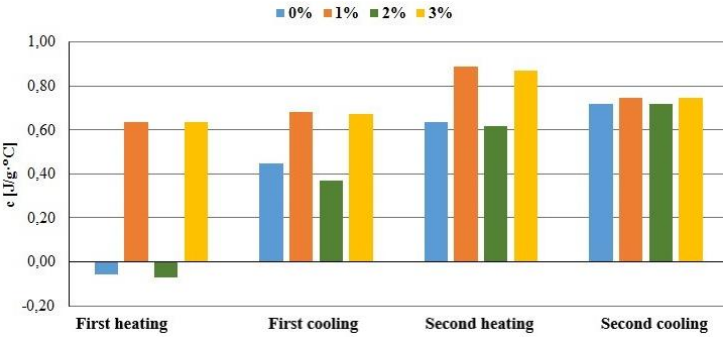

Fig. 5. *Specific heat values of E modified epoxy resin materials (90, 140 ºC temperature interval)*

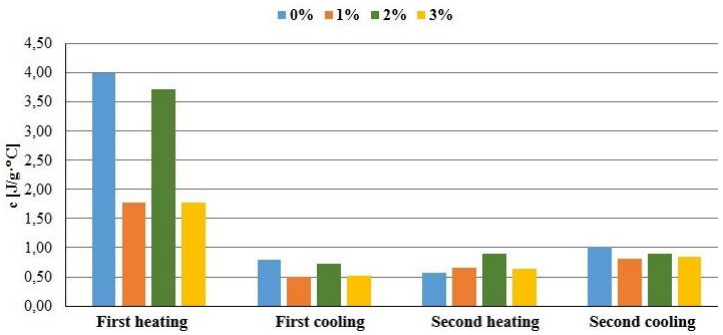

Fig. 6. *Specific heat values of HT modified epoxy resin materials (90, 140 ºC temperature interval)*

Following the analysis of the diagram, we noticed that the highest mass losses were suffered by the materials with epoxy matrix type C and especially with the highest concentration of C3 modifier, presenting the highest mass loss of all the studied materials. The lowest losses were in the case of epoxy type E materials, but of the HT specimens, the most significant was HT0.

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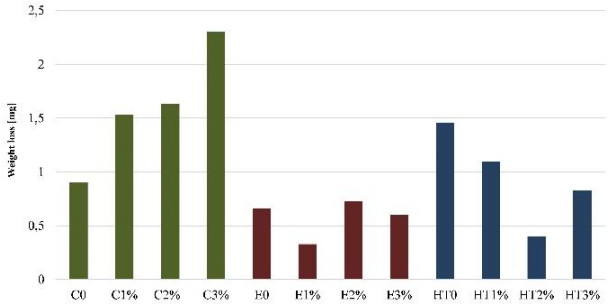

Fig. 7. *Mass loss of polymeric materials*

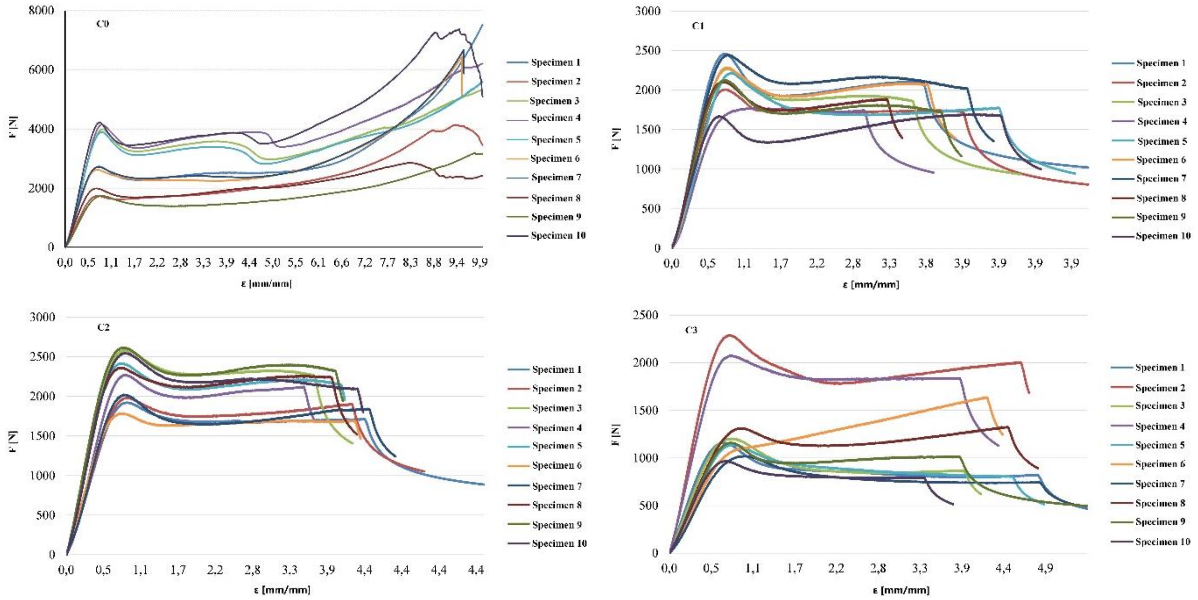

Fig. 8. *Compressive behavior for C materials*

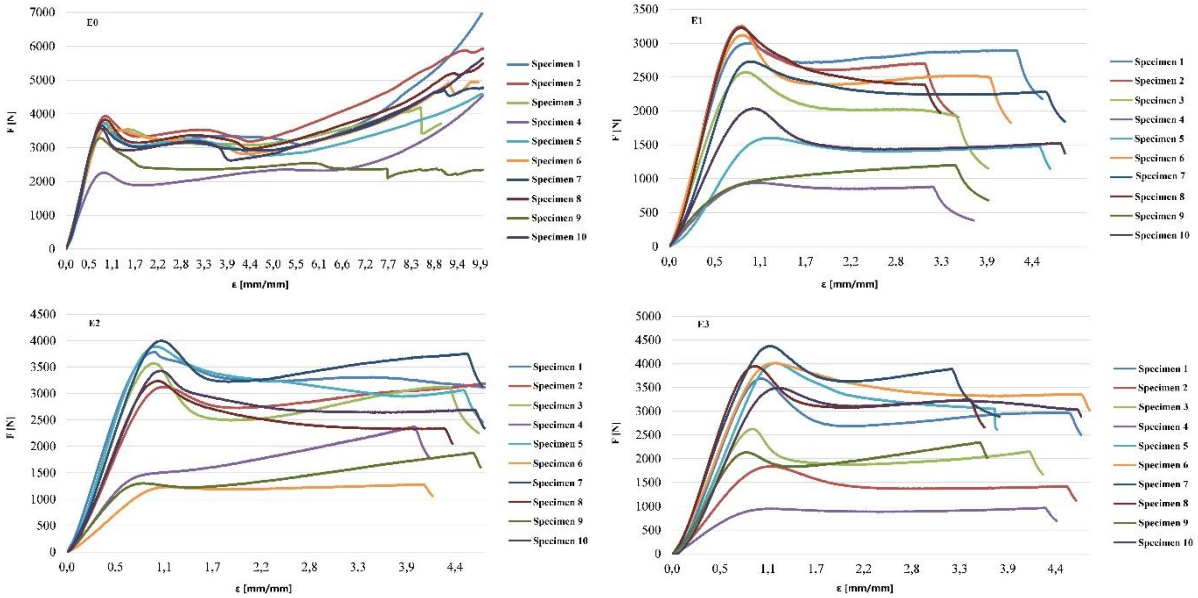

Fig. 9. *Compressive behavior for E materials*

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The compressive behaviour of the formed composite materials is determined by the curves shown in the figure, which show both unmodified epoxy systems of type C and those modified with wheat gluten and gelatin.

From the graphs presented, the unchanged type C epoxy system shows the best mechanical properties at compression, taking into account the value of breaking strength, being approximate twice the value of breaking strength of modified epoxy materials of the same category. In the same curves it can be seen that in the case of C0 type materials, the value of the pressing force was higher compared to the values of the pressing force for the modified materials.

For protein-modified type E epoxy composites, an increase in the degree of compression of the modified materials can be observed compared to the unmodified epoxy system.

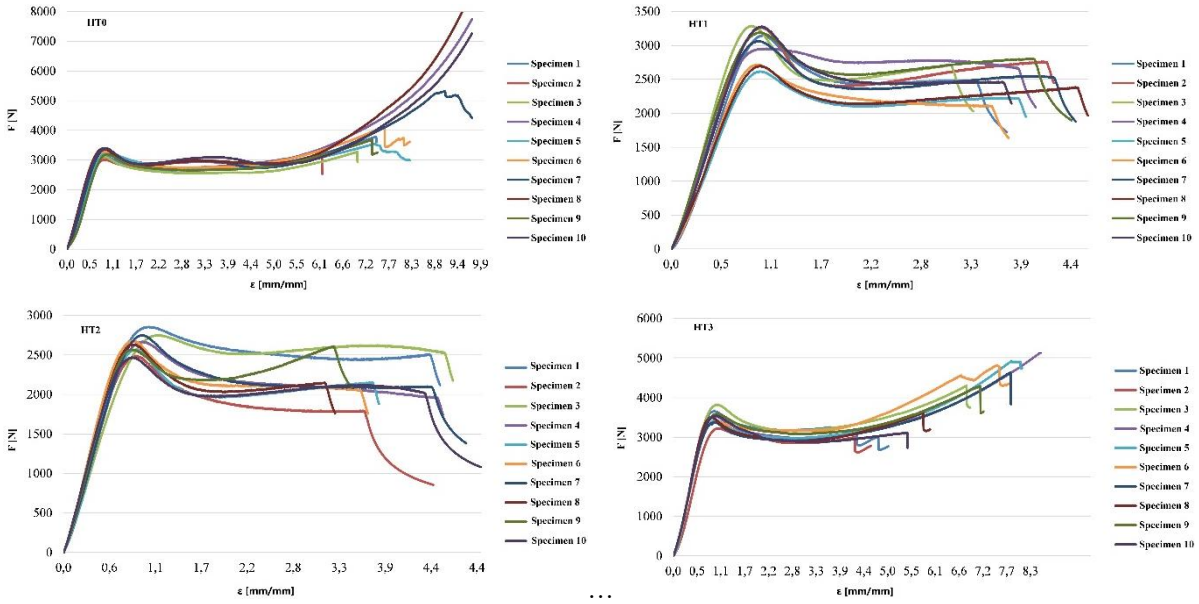

Fig. 10. *Compressive behavior for HT2 materials*

In the case of unmodified and modified HT2 epoxy resin modified with gluten and gelatin, subjected to compression, it can be seen from the drawn curves that the difference between the ten specimens tested is insignificant. Also, if we make a comparison between the curve for the epoxy system and the modified materials, we notice that both the force and the degree of compression are almost identical.

4. Conclusions

Following the analysis of the results, obtained after testing the materials to mechanical testing, it was possible to draw the following conclusions. The use of organic agents to change the properties of epoxy resins leads in particular to increased flexibility as well as ensuring the transport of structures in the volume of the polymer. Following the tests performed we can see that the modifying agents did not significantly change the properties of the formed material, the composite material modified with proteins had a behavior very similar to the unmodified epoxy system. The other parameters were analyzed for this category of materials, which confirms that the concentration of the modifying agent does not significantly influence the properties of the modified materials.

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REMOVAL OF DRUG POLLUTANTS FROM AQUEOUS MEDIA USING CLAM SHELLS WASTE

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ABSTRACT

The pharmaceutical products represent a serious environmental issue in the municipal wastewaters and waste water treatment plants (WWTP). Due to their resistance to biological degradation, the elimination of these pollutants is difficult. In the last years, paracetamol has been detected in higher concentrations in wastewaters. In order to avoid disturbing the aquatic life, economically viable procedures should be identified for removing these types of pollutants. In this context, the present paper has evaluated the efficiency of clam shells waste for retaining paracetamol molecules dispersed in aqueous solutions. The obtained results showed that high removal efficiency is obtained for 60 minutes adsorption duration and a ratio of about 1:100 clam shells: aqueous solution.

KEYWORDS: wastewater treatment, paracetamol, clam shells, adsorption

1. Introduction

The pharmaceutical products represent a serious environmental issue in the municipal wastewaters (MWW) and waste water treatment plants (WWTP). Due to their resistance to biological degradation, the elimination of these pollutants is difficult.

Residual concentrations of pharmaceutical compounds in post-treated MWW can bio-accumulate and cause ecotoxicological damages and indirect acute and chronic toxicity to humans.

In 2018, the European Environmental Agency (EEA) reported that pharmaceuticals are responsible for endocrine-disrupting effects on animals and humans and inhibit photosynthesis or plant growth [1].

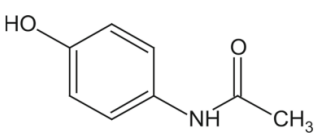

Fig. 1. *Chemical structure of paracetamol (N-acetyl-p-aminophenol)*

The occurrence of pharmaceutical products in MWW may be associated with pharmaceutical and hospital effluents, human and animal excretions, improper disposal of pharmaceutical products, and previously contaminated ground or freshwater used as a primary source for the production of freshwater for human consumption (Table 1).

Paracetamol (PAR) is the priority drug in the aquatic environment based on criteria of toxicity and persistence in the environment (Figure 1) [2].

Paracetamol is commonly disposed into the aquatic environment and can cause hepatotoxicity and nephrotoxicity towards aquatic animals, which eventually results in aquatic death or affects the next level of consumers [2, 3].

In the last years, paracetamol has been detected in higher concentrations in wastewaters. In order to avoid disturbing the aquatic life, economically viable procedures should be identified for removing these types of pollutants.

However, efficient and low-cost methods to deal with the environmental and health problems of paracetamol must be implemented.

Adsorption is extensively used for the removal of PAR from the environment usually at concentrations lower than ppm [4]. Compared to the advanced oxidation process (AOP), adsorption is less costly and less energy consuming. The adsorption can be considered a more advantageous removal method, as it is less complex, easy to apply, and can use cheap, naturally occurring adsorbent materials, even of the biomass waste type.

Activated carbon is one of the best adsorbents to remove common synthetic pollutants from water, and

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was proved to be efficient up to concentrations lower than ppm, which is solely due to its highly developed microporosity and surface area [4].

Clam shells (mussels, clams) have the ability to efficiently remove pollutants from wastewater (metal ions), due to the presence of aragonite, a biogenic form of calcium carbonate, which exhibits ion exchange properties (Table 2).

Table 1. *The concentration of several common analgesic and antipyretic drugs found in the aquatic environment* [3]

Type of drug	Concentration, µg/dm^3			
	Municipal wastewaters	**Treated wastewaters**	**Surface water**	**Drinking water**
Ibuprofen	0.28-6.1	0.11-0.125	0.0045-0.15	< 0.003
Diclofenac	0.46-4.41	0.12-2.89	0.001-0.5	< 0.0025
Paracetamol	45-163	3-16	10-15.5	< 0.298
Salicylic acid	0.9-1.48	0.35-0.47	0.007-0.25	-
Naproxen	0.9-321	0.5-2.4	11-25	-

Table 2. *Physico-chemical characteristics of shellfish waste [5]*

Content of $CaCO_3$, %	Particle size, mm	Volumetric density, g/cm^3	Specific surface, m^2/g
95.6-96.8	0.85-1.18	1.46	2.13

Clam shells contain aragonite, a mineral that has a good ion exchange capacity and can be an alternative to synthetic ion exchangers [5].

In addition, by calcination at 600-800 °C, clam shells have a higher yield in the adsorption of other pollutants as calcium carbonate is converted to calcium oxide [6].

The reactivity of different types of natural calcium carbonates decreases in order: *dolomite* < *chalk* < *marble* < *shell limestone*.

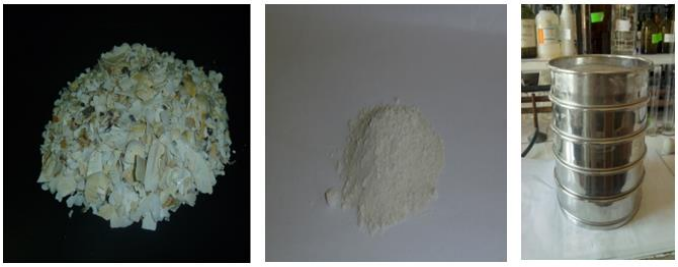
In the present paper, the efficiency of clam shell waste was evaluated for the retention of paracetamol (PAR) pollutants from aqueous solutions.

2. Experimental

2.1. Materials

Clam shells - in powder form with particle diameter between 100 and 250 µm, obtained by grinding for 5 h in a ceramic ball mill with diameter = 1 cm. After grinding, the material was subjected to the dry grading process on the sieves with mesh diameters of 500 µm, 200 µm, 100 µm, 50 µm, and 20 µm (Figure 2) [6].

Para-acetyl-aminophenol (Paracetamol) 500 mg tablets as 0.15 g/L solution in distilled water (PAR).



a) b) c)

Fig. 2. *Clam shells: a) initial state; b) after grinding; c) Granular material sieving equipment*

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2.2 Methods

The particle size distribution of the ground clam shells was determined in the laboratory by a dry grading process on a sieve with a mesh diameter of 500 µm, 200 µm, 100 µm, 50 µm, and 20 µm (Figure 2c).

The static adsorption method is performed by mixing the adsorbent material with paracetamol solution for a specified time and filtering the mixture on filter paper [6].

Determining the content of PAR in solution was performed using the analysis method of oxidizable organic substances. This method consists of analysing the specific parameter for determining the concentration of organic compounds in wastewater as chemical oxygen consumption (CCO). This is a parameter that estimates the degree of chemical oxidisability of compounds dissolved in water. The content of organics was determined by measuring the amount of $KMnO_4$ reduced by oxidizable substances in the aqueous solution, where the excess of potassium permanganate oxidizes organics from water in acidic and high temperature conditions [1, 2].

The pH of the solutions was measured using colorimetric methods.

The retention degree of PAR from the aqueous solution was determined by measuring the concentration of organic substances in the solution before and after the clam shell treatment and was calculated with the formula [6]:

$$\varepsilon = \frac{Ci - Cf}{Ci} x100\,,\ \%, \qquad (1)$$

where: Ci - initial pollutant concentration; Cf - concentration of pollutant after treatment.

2.3 Description of the experimental programme

2.3.1. Determination of the optimal duration of adsorption

Parameters:

- concentration of PAR = 0.15 g/L;
- adsorption ratio ($m_{clamshells}$:V_{sol}) = 1:100 (g:mL): 1 g clam shells powder to 100 mL solution of PAR;
- adsorption duration = 5, 10, 15, 20, 30, 60, 120 minutes.

Working procedure: 1 g of clam shell powder was added to 100 mL of PAR solution (0.15 g/L) using an Erlenmeyer flask. The mixture is shaken for 5, 10, 15, 20, 30, 60, and 120 minutes and then filtered through filter paper (at least 2 samples are taken in parallel for each duration).

Before and after treatment the amount of organic substances in the solution has been analysed.

2.3.2. Ratio optimization $m_{clamshells}$:V_{sol}

Parameters:

- concentration of PAR = 0.15 g/L;
- adsorption duration = 60 min;
- adsorption ratio ($m_{clamshells}$:V_{sol}) = 1.5:100, 2:100, 2.5:100.

Working procedure: 1.5 g, 2 g, 2.5 g of clam shell powder was added to 100 mL PAR solution (0.15 g/L) using an Erlenmeyer flask. The mixture is shaken for 60 minutes and then filtered through filter paper (at least 2 samples are taken in parallel for each duration).

Before and after treatment, the amount of organic substances in the solution has been analysed.

3. Results and discussions

3.1. Particle size distribution of clamshells

It is revealed that these materials have particle diameters between 100 µm and 500 µm (Figure 3).

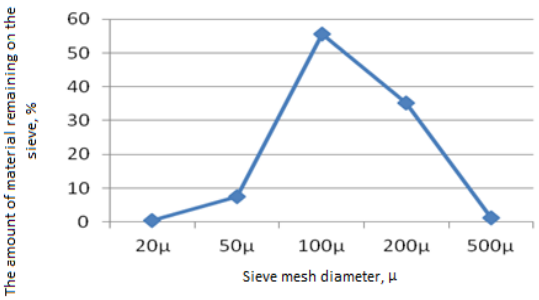

Fig. 3. *The size distribution of clam shells particles*

3.2 Optimal adsorption time determination

The results obtained from the experiments, expressed in terms of adsorption efficiency are presented in Figure 4 as the adsorption efficiency of the organic pollutants on the clam shell substrate versus the adsorption duration. The increase in adsorption efficiency is observed with contact duration of up to 60 min. After this duration, the adsorption efficiency worsens. This can be explained by reaching adsorption equilibrium when the adsorbent material is exhausted and needs to be regenerated. In Figure 5, it can be observed that at the same contact time the maximum amount adsorbed by 1 g of clamshells is 148.52 mg of organic substance.

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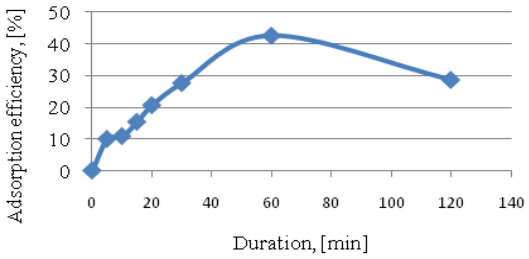

Fig. 4. *The influence of contact time on the efficiency of the organic pollutants' adsorption*

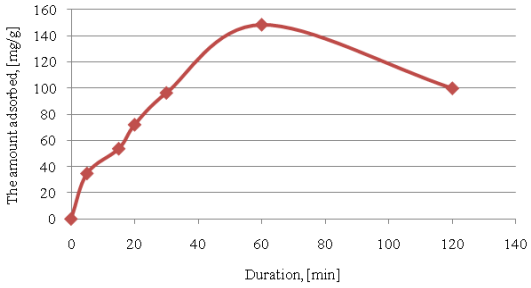

Fig. 5. *The influence of contact time on the amount of organic pollutants adsorbed by 1 g of clam shells*

3.3. Ratio optimization $m_{clamshells}$:V_{sol}

The results obtained under the conditions of the parameters presented in 2.3.2 are shown in Figures 6, 7 where it can be seen that the best adsorption efficiency of organic substances was obtained at a ratio $m_{clamshells}$:V_{sol} of 1:100.

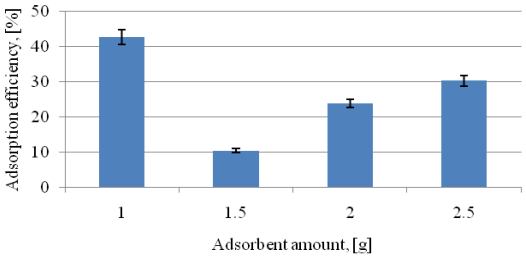

Fig. 6. *The influence of the adsorbent quantity on the adsorption efficiency*

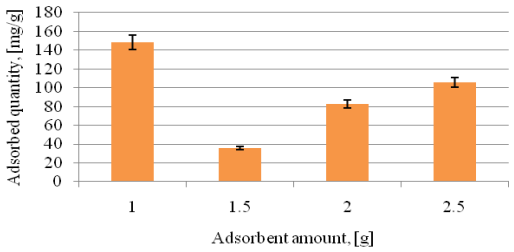

Fig. 7. *The Influence of the quantity of adsorbent on the quantity of organic pollutant retained*

4. Conclusions

The obtained results on the evaluation of paracetamol removal from aqueous solutions using clam shell waste as adsorbent material led to the following conclusions:

- The static adsorption used to retain paracetamol on clamshell waste particles resulted in high yields for an adsorption time of 60 minutes and a ratio of $m_{clamshells}$:$V_{solution}$ of 1:100.
- The method uses cheap and available adsorption materials (biomass waste) and can be an alternative to advanced oxidation methods for the removal of drugs from wastewater, which are relatively expensive and complex and have a negative impact on the environment.
- The future research will be aimed at the study of all factors that could influence the process as well as the study of adsorption isotherms and process kinetics in order to determine the exact conditions of using these wastes in the process of organic pollutant retention from aqueous media.

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