

**Republic of Iraq
Ministry of Higher Education &
Scientific Research
University of Babylon
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Department of Physics**



**Effect of Egg Shell Powder on Damping and
Swelling Properties (Rubbery Blends/Carbon black)
in Nano composite**

Research

**Submitted to the council of the College of Education for Pure Sciences of
University of Babylon in Partial Fulfillment of the Requirements for the
Degree of Higher Diploma Education / Physics of Materials and its
Applications**

By

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2023 A.D

1445A.H

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ
يَا أَيُّهَا الَّذِينَ آمَنُوا اتَّقُوا اللَّهَ حَقَّ تَقَاتِهِ لَعَلَّكُمْ تُفْلِحُونَ

اللَّهُ نُورُ السَّمَاوَاتِ وَالْأَرْضِ مِثْلُ نُورِهِ كَمِشْكَاةٍ
فِيهَا مِصْبَاحٌ الْمِصْبَاحُ فِي زُجَاجَةٍ الزُّجَاجَةُ كَأَنَّهَا
كَوْكَبٌ دُرِّيٌّ يُوقَدُ مِنْ شَجَرَةٍ مُبَارَكَةٍ زَيْتُونَةٍ لَا
شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ
تَمْسَسْهُ نَارٌ نُورٌ عَلَى نُورٍ يَهْدِي اللَّهُ لِنُورِهِ مَنْ
يَشَاءُ وَيَضْرِبُ اللَّهُ الْأَمْثَالَ لِلنَّاسِ وَاللَّهُ بِكُلِّ شَيْءٍ

عَلِيمٌ

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ
يَا أَيُّهَا الَّذِينَ آمَنُوا اتَّقُوا اللَّهَ حَقَّ تَقَاتِهِ لَعَلَّكُمْ تُفْلِحُونَ

سورة النور الآية (39)

Dedication

Thanks go to Allah and at all the whole grace prayer, and peace upon the best creation Mohammad (peace be upon him and his family) and his divine good...

To the spirit of my father and my mother are the source of light in my life

To my wife and children, the source of love and affection.

To my teachers the lighted candles who are showing us the road of life.

To my close friends

To My country with honour and dignity and everyone who helped me.

Acknowledgments

Praise be to Allah Lord of the world, and best prayers and peace upon his best messenger Mohammed, his pure descendants, and his noble companions.

Then, I would like to express my deep gratitude and appreciation to my supervisor, Prof .Dr. Sameer Hassan Hadi AL-Nesrawy for his guidance, suggestions, and encouragement throughout the research work, without him, this work would have been impossible.

I would like to thank the staff of the department of physics in college of education for pure sciences in the University of Babylon for their kind attention and assistances.

Finally, I would like to thank all members of my family for their help and encouragement and all who helped me during the preparation of this dissertation

falah 

Supervisor's Certification

I certify that this thesis entitled "**Effect of Egg Shell Powder on Damping and Swelling Properties (Rubbery Blends/Carbon black) in Nano composite**"

Is prepared by the student (**Falah Shaker Abdulzahra**) under my supervision at the College of Education for Pure Sciences, University of Babylon in partial fulfillment of the requirements for the Degree Higher Diploma Education / Physics of Materials and its applications

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Date: / / 2023

Examining Committee Certification

We certify that we have read this thesis entitled "**Effect of Egg Shell Powder on Damping and Swelling Properties (Rubbery Blends/Carbon black) in Nano composite**"

and as committee, examined the student (**Falah Shaker Abdulzahra**) its contents and that in our opinion it meets the standards of a research for the Degree of Higher Diploma Education / Physics of Materials and its applications.

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Abstract: -

This research overlapping rubber composite batches consisting of Natural Rubber (50NR) and Acrylonitrile Butadiene Rubber (50NBR) as a matrix material and the egg shell powder was added (ESP) and granular size less than (222 μm) and with loading ratios (0,10,30 and 50 pphr) also , carbon black material (C.B) as a support material and with loading ratios (20pphr). Where two groups of composites were obtained:

Group A consists of (50NBR/50NR/ESP).

Group B consists of (50NBR/50NR/ESP/CB–20pphr).

Some mechanical properties such as hardness have been studied, as they were measured using a device (shore A) and according to the specification ASTM D1415 and Swelling ratio for these rubber composites according to the specification ASTM D471 – 98 as well as damping properties such as Rebound Resilience and damping time using a device (Trips meter) by the specification Dunlop trips meter equipment.

The results showed a slight increase in the hardness values with an increase in the loading ratio for the two groups (A and B), and increase is more for the group (B). The results also showed that there is a clear decrease in the values of Rebound Resilience and damping time with an increase in the loading ratio (ESP) for both groups, and this decrease increases with respect to group (B). With respect to swelling with salt water, it was found that these superposition have a slight increase or swelling a slight increase when immersion in saline water.

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List of Abbreviations

NR	Natural Rubber
NBR	Acrylonitrile Butadiene Rubber
SR	Synthetic Rubber
EPDM	Ethylene-Propylene Diene Terpolymer
pphr	Per part hundred Rubber
C.B	Carbon black
DBP	Dibutyl Phthalate Absorption
ASTM	American Society for Testing Material
MBS	N-oxydiethylenebenzothiazole
Tg	The degree of glass transition
FCC	catalytic cracking tar
ESP	egg shell powder

List of Symbols

symbols	meaning
t_0	Initial thickness
t	Final thickness
N	Load in Newton
P	depth of Penetration
Δ	Logarithmic Decrement
ζ	Damping Ratio
ψ	Specific Damping capacity
δ	phase Angle
Q	Quality Factor
C_c	Critical damping coefficient
M_A, M_B	This masses
V'_A, V'_B	velocities after impact
F	indenting force (N)
k	Young's modulus (MPa)
r	radius of the indenter (mm)
P	depth of Penetration (mm)
ρ_p	mass density of sample
ρ_t	mass density of solvent
W_i	initial weight of the sample
w_s	weight of swollen sample
V_v	volume swelling ratio

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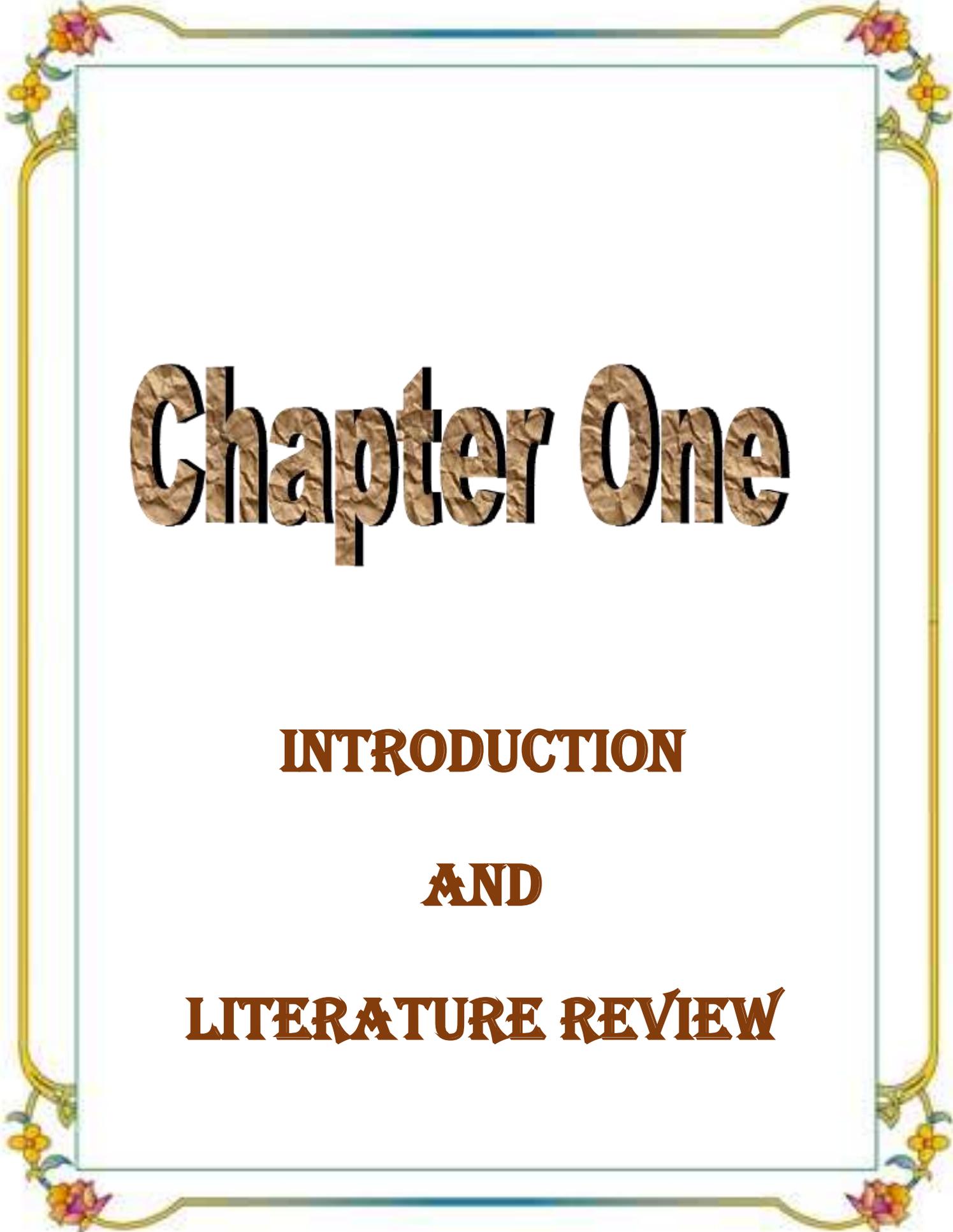
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A decorative border with a gold-colored frame and floral motifs in purple, yellow, and green at the corners.

Chapter One

INTRODUCTION

AND

LITERATURE REVIEW

1-1 Introduction

The growing use of rubber in engineering applications results from its unique properties that include high extensibility, high strength high – energy absorption, and high resistance to fatigue. Other attributes are good environmental resistance and high resilience. Hence, ability of rubber to undergo very high strains without permanent deformation makes it ideal for many applications, Flexible polymers (rubber) contribute greatly to the contemporary development that we live in our daily lives. Rubber is the basis for the manufacture of tires for cars and aircraft, which in turn contribute to the movement of transport and communication. Most types of rubber are complementary parts of many industries, which are difficult to list, starting with gums. And specifications and ending with the electrical industries and rocket assemblies for space flights[1].

1.2 Natural Rubber (NR)

The history of the discovery of Natural rubber date back to the eleventh century. Man used to paint his clothes to prevent them from getting wet. I made Rubber balls from him to play, but the name rubber was launched by the scientist (Priestley) in the year (1770 AD), as noticed that it au's able to. Erased the traces of writing the pencil and noticed that the trees are able to form this rubber material [2] NR is only natural product. It is a cis- poly isoprene but contains a variety of impurities such as proteins and resins. It crystallizes much more readily upon stretching compared to synthetic cis-poly isoprene, IR, which dos not contains long

branches [3] Natural rubber is collected in the form of latex that exudes from the bark of the tree when it is cut. The tree *Hevea Braziliensis* is the largest producer of this latex. The average rubber content of latex may range between 30- 45%.

Latex concentrates are differentiated by the method of concentration and type of preservative used. Concentration is achieved by centrifugation (most common), by creaming, or by evaporation [4]. The elastomer, used alone or with reinforcing filler, possesses first class mechanical properties with excellent process ability. It crystallizes easily at low temperatures and, so in sub-zero temperatures, it is necessary to it by storing in a hot room at $40 - 50\text{ C}^0$ (before winter season both these operations are unnecessary with synthetic elastomers . NR requires lower curing temperatures and therefore longer vulcanization time. At mixing, NR has a sufficiently high viscosity to allow optimum dispersion of reinforcing fillers and other ingredients [5].

Chemical Structure Natural rubber is a high molecular polymer isoprene, C_5H_8 . The repeating unit is $(-\text{CH}_2 - \text{C}(\text{CH}_3) = \text{CH} - \text{CH}_2 -)_n$. The Hevea rubber is the major naturally occurring form of cis 1,4- poly isoprene. This rubber contains more than 98% of its double bonds in the cis – configuration that is essential for elasticity in poly isoprene. Over 90% of all cis 1,4- poly isoprene used industrially is natural Hevea rubber[6]. The double bond in each repeating unit in the polymer chain is a site of steric isomerism since it can have either a cis or a Trans configuration. The polymer chain segment on each carbon atom of the double are located on the same side of the double bond in the cis configuration and on the opposite sides in the trans configuration, see figure (1.1)[1]

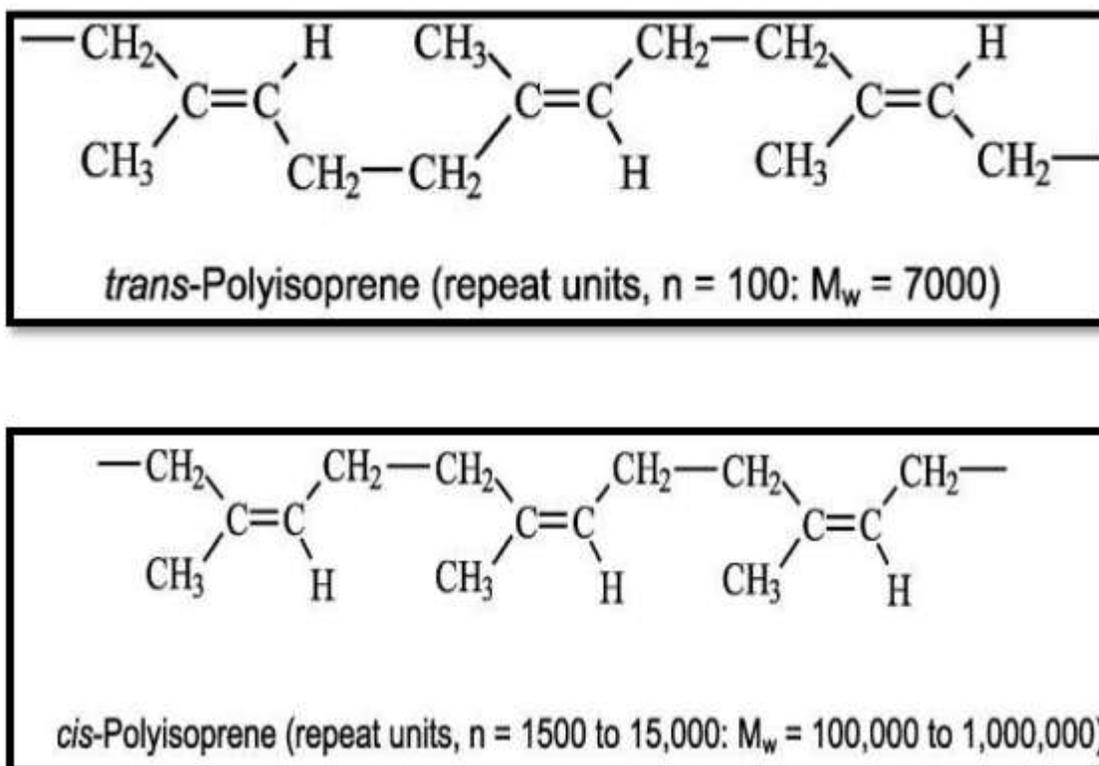


Figure (1.1) cis and Trans- isomers of natural rubber [7].

The Trans isomer which has a higher molecular symmetry crystallizes to a greater extent and therefore has higher melting temperature T_m and the degree of glass transition T_g Values and significant crystallinity. It is less an elastomer and more like a thermoplastic. The T_g of NR is about 200 K its T_m equal to 298 K and its structure is thought to be completely cis 1,4- poly isoprene, except for the chain ends , 1,4- poly isoprene is found in both the cis and trans form[8]

1.3 Acrylonitrile Butadiene Rubber (NBR)

Nitrile rubbers (NBR) are copolymers of butadiene and acrylonitrile with the proportions ranging from 55% to 82% butadiene and 18% to 45% acrylonitrile. These rubbers are more costly than ordinary rubbers, Also known by the name Buna-N, it was first founded in Germany at 1937 by I.G. Farbenindustrie . The structural formula of NBR is as shown in Fig. (1.2)[9]

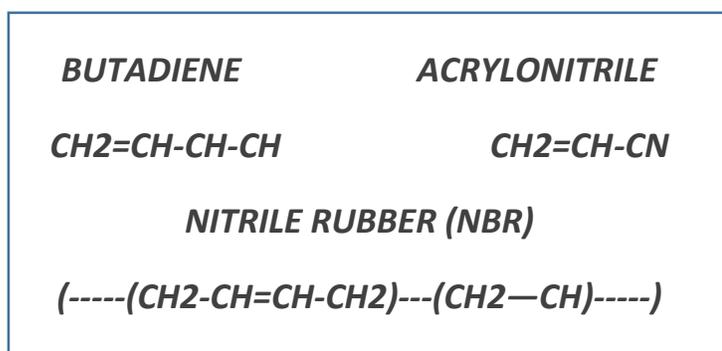


Fig (1.2) structure of NBR

Nitrile Rubber is commonly considered the workhorse of the industrial and automotive rubber products industries.(NBR)is actually a complex family of unsaturated copolymers of acrylonitrile Nitrile Rubber (NBR). (NBR) is a complex family of unsaturated copolymers of acrylonitrile and butadiene. By selecting an elastomer with the appropriate acrylonitrile content in balance with other properties, the rubber compounder variety of application areas requiring oil, fuel, and chemical resistance. In the automotive area, (NBR) is used in fuel and oil handling hoses, seals and grommets, and water handling applications.[10]

With a temperature range of -40°C to $+125^{\circ}\text{C}$, NBR materials can withstand all but the most severe automotive applications. On the industrial side NBR finds uses in roll

covers, hydraulic hoses, conveyor belting, graphic arts, oil field packers, and seals for all kinds of plumbing and appliance applications. Worldwide consumption of (NBR) is expected to reach 368,000 metric tons annually. [11]

1.4 Rubber composites

Engineered rubber products consist of all rubber, or rubber combined with other materials. Product complexity ranges from that of a simple rubber band to complex composites such as radial tires or rubber-metal bearings for use in aerospace applications [12]. Materials such as steel, aluminium, plastics, fabric, and cords are often combined with rubber to form composites. The purpose of these materials is generally to increase strength, minimize distortion [12]. Rubber compounds exhibit several phenomena like the ability to retain elastic properties during prolonged action of compressive stresses compression set behavior, and loss of resiliency. The lower the percentage of compression set, the better the material resists permanent deformation under a given deflection and temperature also, the selection of rubber compounds for use in engineering industry [13]. High elastic polymer composites are very important in the applications of rubber industries, such as tires transportation belts, pipes for fluids and oils transmission, damping and support parts in the mobiles as well as diaphragm. Rubber composite materials with different type of rubber are used in dampers and supports application. Therefore: it is occupied a wide field studies, because rubber has good characterization in the high elastic strain damping which is obtained from the suddenly impact loading because of different acceleration of the system. Therefore a different modification of new type of rubber composite for loading resistance in addition to the interference of the usages conditions with the

mechanical loads such as environment effects at high temperatures in the presence of oils and friction result in creation of blended polymers such as (NBR, SBR , NR , CR ,etc) [7,14]

1.4 Carbon black (CB)

A carbon-containing substance that results from the incomplete combustion of products containing hydrocarbons such as heavy derivatives of petroleum such as catalytic cracking tar (FCC) and coal tar. Carbon black is a form of amorphous carbon.

Carbon black is used as a pigment in the manufacture of plastics, paints and inks. It is also used as a filler in the manufacture of tires and other rubber products.

Carbon black is second only to rubber in terms of importance in the rubber industry, Being a good strengthening agent and appropriate, It helps in improving the physical and mechanical properties of the rubber compound used in the industries inflatables [15]

Carbon black is the predominant reinforcing filler used in rubber composite

Improved toughness and strength. For different types of tyres, Carbon black is also used in molded synthetic rubber products, Such as belts, hoses, vibration isolation devices, bumpers, various types of cushions, shoes, and car tires [16].

1.5 Previous Studies

There are many studies that investigate the rubber recipes contents, the function of all ingredients, and their effects on damping properties such as Resilient, Hardness, etc...

Typical studies are presented as follows:

Al-Maamory (2003) [17], Studied the effect of humidity on the properties of natural rubber (SMR 20) and synthetic rubbers (SBR 1502) and (BR-cis)

Rattanasom (2005) [18], were elucidated the effect of a sulfur vulcanization system, i.e. conventional vulcanization (CV) and efficient vulcanization (EV) on the mechanical properties and heat aging resistance of natural rubber (NR)/tire tread reclaimed rubber (RR) blends.

Agarwal (2005) [19], used Scanning Electron Microscopy (SEM) analysis of tearing of natural rubber (NR) vulcanizes has been carried out with respect to, (1) nature of crosslinks, (2) extent of crosslinking, (3) effect of addition of reinforcing carbon black filler (HAF N330), and (4) effect of temperature, as obtained in the SEM photographs of the vulcanizates subjected to tear fracture.

Yi Li (2007) [20], investigated a process for the rotationally molded truck fender was studied. The relationship between the part's impact strength and the heating temperature and time was analysed. Furthermore, the influence factors for the impact strength were described by investigating the melt flow index (MFI) in the sample. Finally, the optimized process conditions for a truck fender were given.

Al-Masudi (2007) [21], prepared five raw rubbers (NR, BR-cis, SBR, IIR and BIIR), two blends (SBR: NR and IIR: BIIR), five loading levels of carbon black (type N-375) with SBR and finally six percentages of LDPE with SBR alone and with IIR alone. So the damping measurements have been carried out by two methods: Free Vibration method and resilience pendulum method

Suleiman (2007) [22], studied the effect of adding the Silica filler with amounts(25,50,75,100)pphr Styrein Butadiene (SBR), on the mechanical properties and homogeneity of the SBR was studied. It was the found that the amount 50 parts of Silica per 100 parts of SBR gave best compound with high homogeneity and best mechanical properties with the condition that it should be added to the rubber in its elastic stage and not in its glass stage.

AL-Noumannee (2010) [8], investigated the effect of the different blending of SBR with NR, with different particle size of carbon black and blending with silica filled NR /SBR blend and the effect of carbon black silica interaction and the sulfur content on the mechanical properties of composites.

AL- Nesrawy S.H(2014)[23] ,studied finding alternatives for the material basis supported and the material used in the preparation of the fenders, which were prepared and manufacture Ajnat rubber consisting of synthetic rubber styrene butadiene rubber (SBR 1502) and synthetic rubber BRcis addition to natural rubber NR SMR 20)) as materials.

Farzadq (2016) [24] , studied the effect of the molecular size of carbon black (N326 ,N375 N660) on the properties (Rebound Resilience , damping time) and the mechanical properties (hardness , Specific Gravity) of synthetic rubber (BRcis) known as BONA. (Hardness) And (Specific Gravity) tests have shown that the carbon black rubber (N326) is more solid and has a specific gravity than (N660, N375). Likewise, tests (Rebound Resilience) and (damping time) showed that the rubber reinforced with black carbon (N326) is less Rebound Resilience and has a damping time of both types (N660 , N375).

Muhammad Juma'a (2019) [25], studied the rheological properties (density and specific weight) as well as mechanical properties such as (ultrasound velocity, relaxation time, relaxation capacity, vocal impedance, compression and elastic modulus) of rubber compounds containing (NR / SBR / OSP) Where he prohibited without carbon black (CB) once and by using carbon black again with loading ratios (CB) .

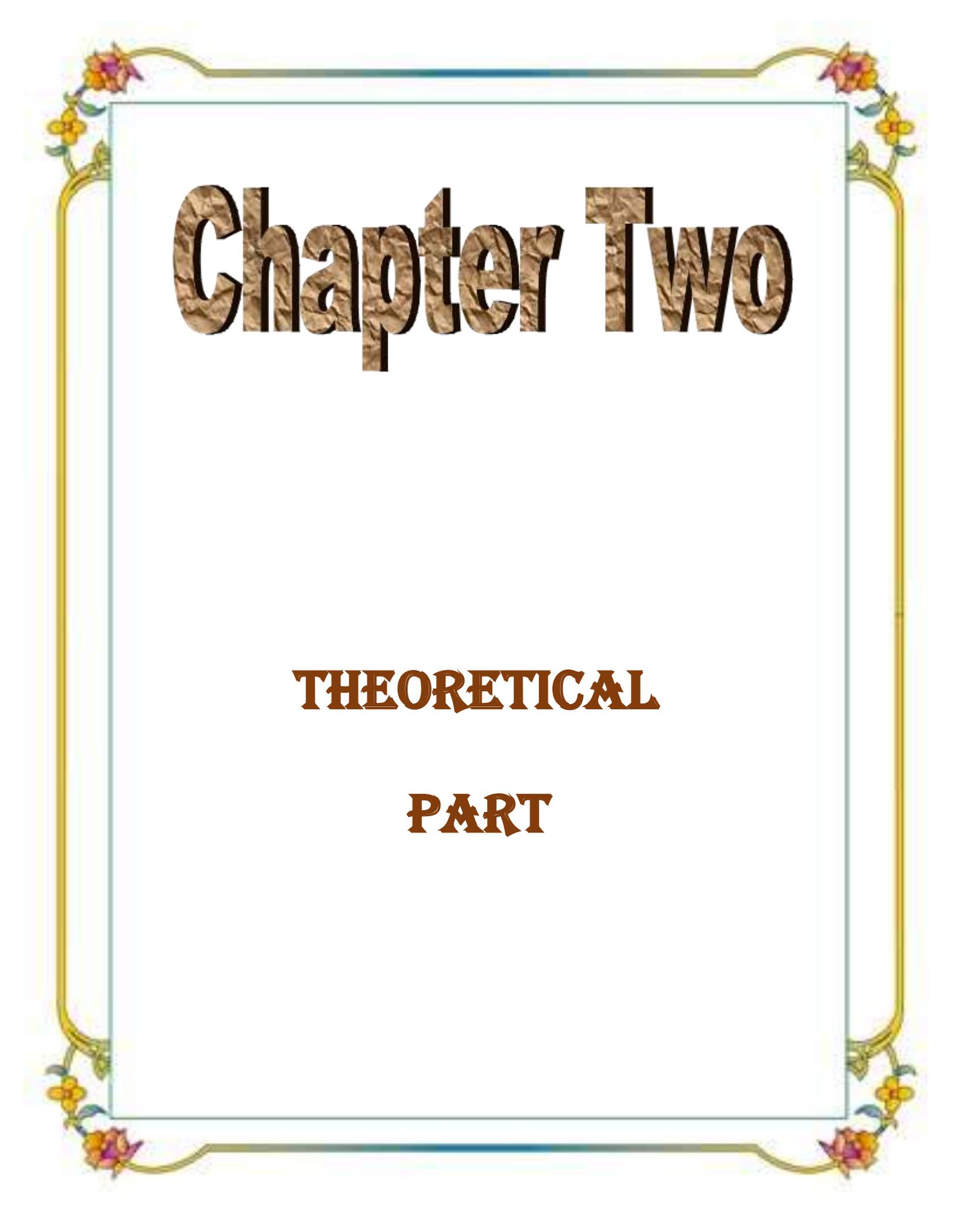
Mousa Hawan Naeem (2019)[26], Preparation of (Rubber Blend/Oyster Shell Powder) Composites and Study Rheological Properties. Decreasing the results of all these properties with increase the loading ratio of OSP with or without C.B 10 pphr. Bustan fadel abud (2020) Study the Damping properties and swelling in saline water for (NR 50 / SBR 50 / C B / OSP) composite.

Essam Filayyih Al-Nesrawy (2021) [27] studied the Rheological Properties Of (NR / SBR / CMC / C.B) Nano composites .

1.6 The aim of this research

1-Preparing the (50NBR/50NR/ESP) and (50NBR/50NR/ESP-CB20) Composites.

2- Investigation of the effect of egg shell powder (ESP) on the damping and swelling in saline water for (50NBR/50NR/ESP) and (50NBR/50NR/ESP-CB20) Composites.



Chapter Two

THEORETICAL

PART

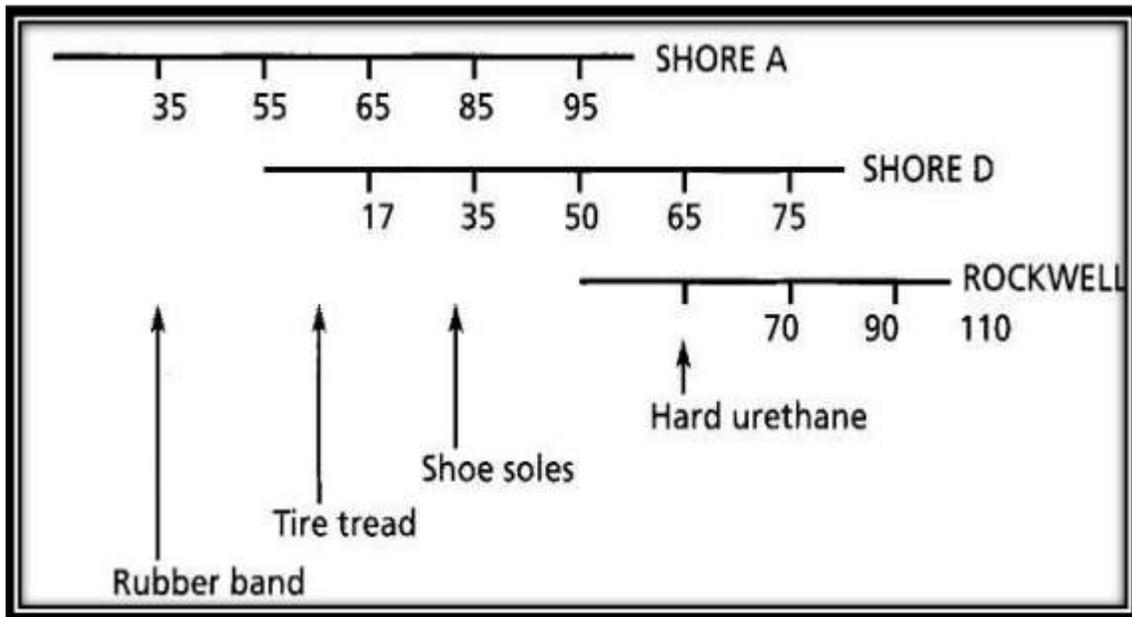
2.1 Introduction

Rubber is similar to those materials consisting of long polymer chains with a high degree of elasticity and mobility, which are interconnected in a network structure. The requirements for flexibility and mobility are associated with a very high susceptibility to deformation. As a result of externally imposed stress, long strings may change their configurations, a modification that occurs relatively quickly due to the string's high mobility. Rubber can transfer to thermoplastic materials. Thermoplastics are three-dimensional molecular networks, with extended molecules held together by chemical bonds. These materials absorb solvents and swell, but do not dissolve in them. However, it cannot be reprocessed once heated. On the other hand, rubber particles are not bonded to heat by primary chemical bonds [28]. Instead they are linked by the physical aggregation of fragments of molecules into solid spheres. Whereas, thermoplastic rubbers dissolve in suitable solvents and soften upon heating so that they can be repeatedly processed for many times. The term “rubber” has been used for any material that when subjected to an external force deforms with a relatively low load or deflection ratio and recovers its original shape quickly and forcibly when the applied forces are removed. Based on this definition, there are several materials that can generally be classified into natural rubber and synthetic rubber [29]. Rubber technologists previously focused on reducing compound cost to lower product costs. Greater emphasis is now being placed on reducing product weight, reducing and even eliminating processes, and combining rigid and flexible materials in new ways to reduce costs. These considerations apply to both framed and non-framed products [12]

2.2 mechanical properties

2.2.1 Hardness:

Perhaps the most widely used test in the rubber industry is the measurement of hardness. Hardness is defined by ASTM as the resistance of material surface to indentation as measured under specified conditions. Rubber technologists use hardness as a convenient means of classifying rubber materials. There are many different hardness tests that claim nearly universal acceptance: the durometer and the International Rubber Hardness tester. Different durometers shown in figure.



Figure(2.1) Approximate equivalence of hardness scales

Which designed to cover the wide range of hardness encountered in rubbers. The shore A scale is the most appropriate for rubber compounds commonly used in engineering. The shore D scale is appropriate for harder rubber compounds such as ebonite, plastics. The Rockwell scale is also used for hard urethane [30]

The International Rubber Hardness tester differs from the durometer in several important aspects. It is a small bench top instrument that uses deadweight's to apply defined loads

to a spherical indenter, while the penetration is measured with a dial gauge. The dial gauge is commonly calibrated directly in International Rubber Hardness degrees (shore A). The shore A scale was chosen to have readings numerically equivalent to the popular A scale over the range normally encountered in practical engineering compounds. While the approximate equivalence of the (shore A) and the A scales is valid for highly elastic materials, significant differences may be expected for materials that show marked time-dependent behaviour [4, 28]

According to the ASTM standard the penetration of the indenter bears a known relation to the Young's modulus of the rubber .This relation is an empirical relation obtained by Scott 1948[30]

$$\frac{F}{E} = 1.9 P^{1.35} r^{0.65} \dots\dots\dots (2.1)$$

Where F is the indenting force (N), E is Young's modulus (MPa), P is the depth of Penetration (mm) ,and r is the radius of the indenter (mm)[30]

Showed that a theoretical relation may be derived from the classical theory of elasticity in the form:

$$\frac{F}{E} = 1.78 P^{1.5} r^{0.5} \dots\dots\dots(2.2)$$

Young's modulus to be equal to three times the shear modulus G, may write:

$$\frac{F}{G} = 5.33 P^{1.5} r^{0.5} \dots\dots\dots(2.3)$$

These relations between hardness and Young's or shear modulus, whether empirical or theoretical, should be regarded as only approximate. Significant discrepancies may occur

Simply from the way Young's modulus is defined and measured hardness. Discrepancies are also expected when the material deviates markedly from perfectly elastic behaviour [28].

2.3 damping properties

2.3.1 Resilience

This property is one of the outstanding features of rubber. It shows the ability of a rubber vulcanized to return the energy used to deform it. Resilience is important mainly because it affects the operating temperature of a rubber product, thus influencing its strength and ageing behaviour .Rebound resilience increases with temperature [31].

Resilience is the ratio of the energy returned upon recovery from deformation to the energy required to produce the deformation [28].

The study of impact strength of material is considered as the first key to study the properties of the material and it is defined for un notched sample as the absorbed energy during the impact to the cross section area of the sample at the fracture of

Polymer of high toughness has high crack energy, and it is possible to calculate the impact strength from the following relation [31].

Impact strength = Energy required for crack / sample cross sectional area. Its unit is (j/m^2) and it depends on various variables including type of the materials, temperature, stress System, strain rate, manufacture, and geometry of article, fabrication and environment conditions. In case of rubber the case is different, when rubber is deformed, the energy which is not returned as mechanical energy is dissipated as heat in the rubber. The ratio of the energy returned to the energy applied is termed the resilience. A collision between two bodies which occurs over a short interval of time is known as an impact.[14]

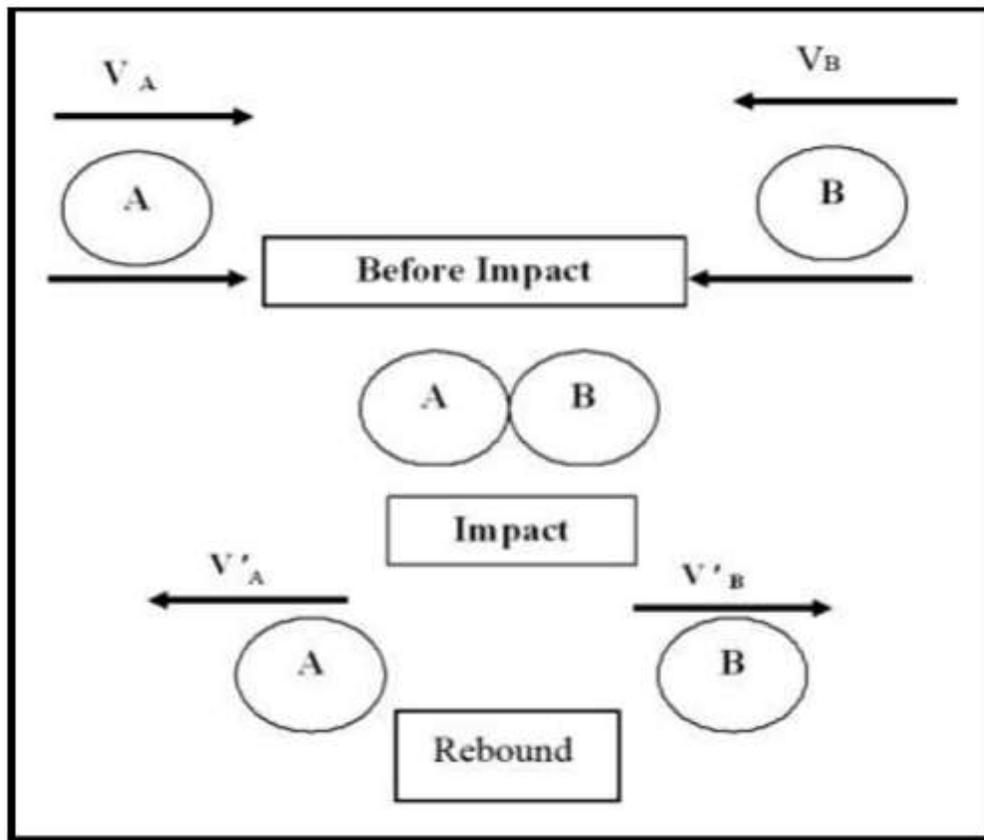


Figure (2.2) Elastic collisions

Consider an impact between two bodies, A and B as shown in Fig. The principle of conservation of linear momentum is used in cases where impacts occur. [14, 32]

$$\text{Momentum before impact} = \text{momentum after impact}$$

$$M_A V_A + M_B V_B = M_A V'_A + M_B V'_B \dots\dots\dots(2.4)$$

Where M_A and M_B are the masses, V_A and V_B the velocities before impact and V'_A , and V'_B , the velocities after impact.

The ratio of the relative velocities is known as the coefficient of restitution (er):
 $e_r = - \text{relative velocities after impact} / \text{relative velocities before impact}$
 $e_r = - (V'_A - V'_B) / (V_A - V_B) \dots\dots\dots(2.5)$

$$e_r = \sqrt{2gh'} / \sqrt{2gh} = \sqrt{\frac{h'}{h}} \dots\dots\dots(2.6)$$

h' / h is known as rebound resilience (for single impact) where h' is the height rebound and h is the height of fall rebound, resilience is related to rebound angle and angle of drop[21]

$$\text{Rebound resilience (R \%)} \text{ uncorrected} = (1-\cos i) / (1-\cos j) * 100\% \dots\dots(2.7)$$

Where i = rebound angle, j =angle of drop (45^0)

Then from equation (2.7)

$$R\% = 341.421 (1-\cos i)$$

$$(R \%) \text{ corrected} = (1-\cos \{i+i.x/2\}) / (1-\cos \{j+j.x/2\}) \times 100 \dots\dots\dots(2.8)$$

Where $x = 1/2n \times \log_e j/i$

n= number of swings The Healey pendulum springs from predetermined height and rebounds to height determined by the resilience of the rubber specimen as shown in Fig. (2.3)

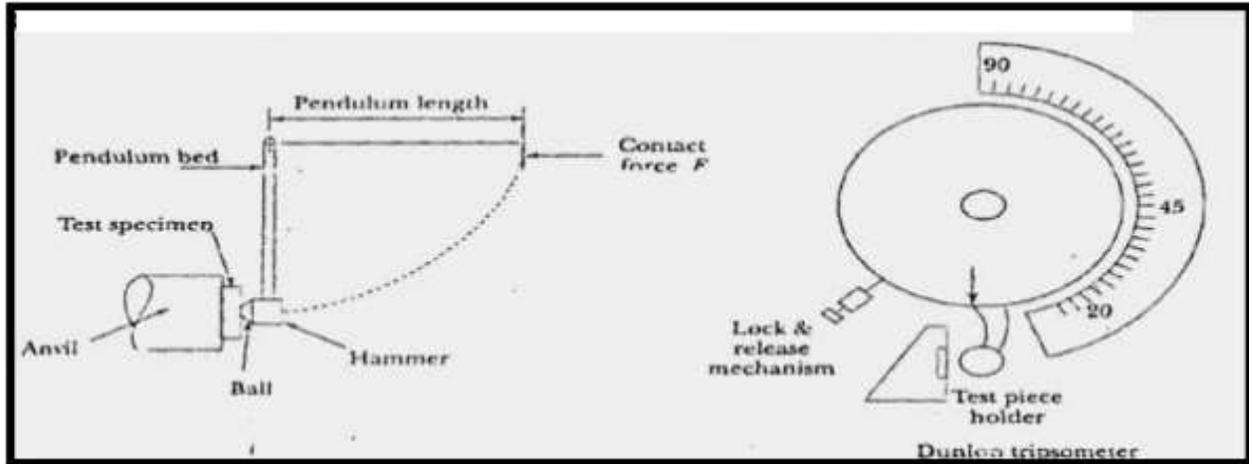


Figure (2.3) Schematic representation of a rebound pendulum

2.3.2 Damping time

With regard to the test samples (NBR50%/ NR50%/ ESP), the damping was chosen for the eight samples in the presence and absence of carbon black, and with different loading rates for eggshell Powder (0, 10, 30, 50), and it was found that the damping time decreases with increasing the level Of loading. The reason for this is due to the increase in the hardness of the samples when the loading percentages are increased, which is inversely proportional to the elasticity.

2.4 Swelling ratio

The action of a liquid on a vulcanized rubber generally results in: (a) absorption of liquid by the rubbery diffusion; (b) adsorption of soluble constituents

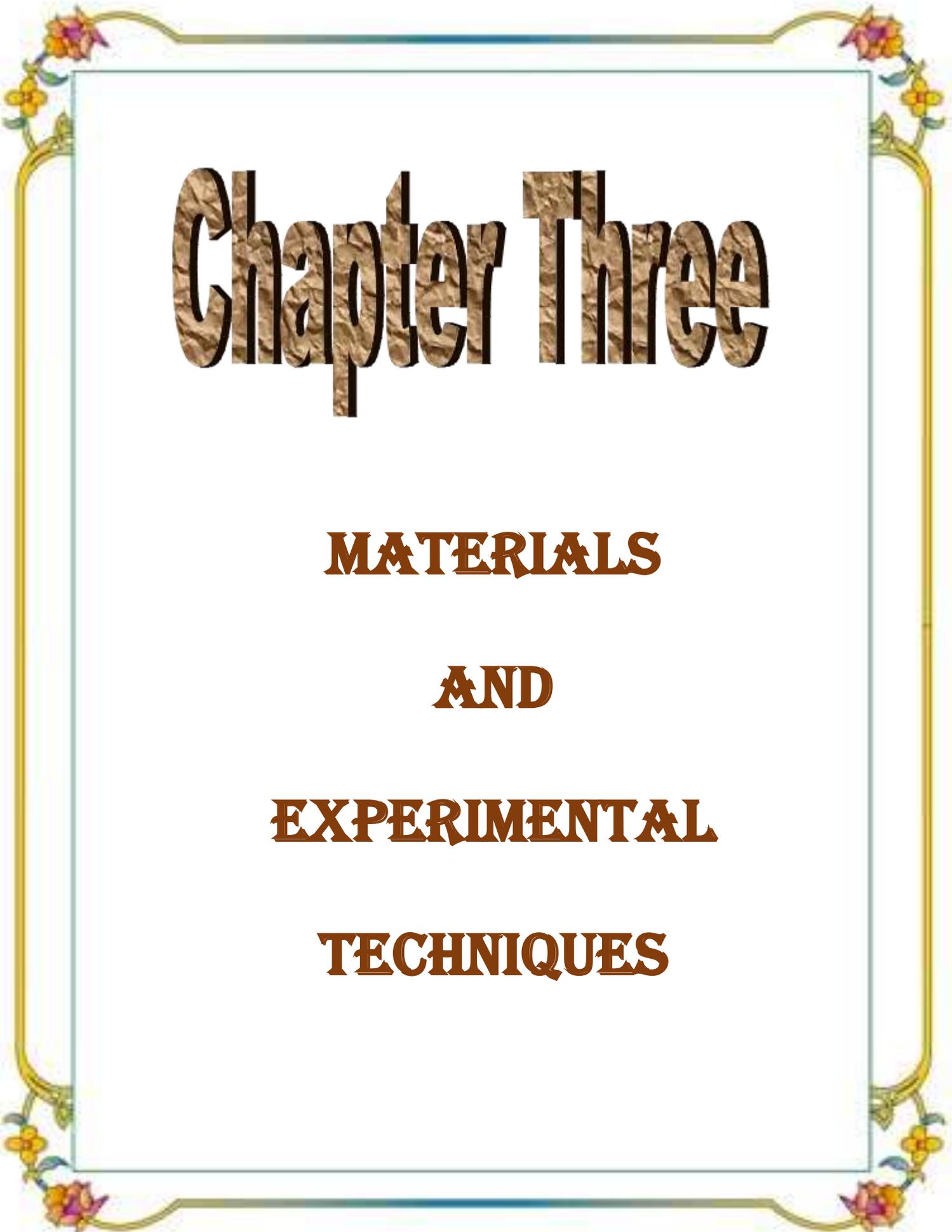
from the rubber by diffusion, evaporation of the liquid and shrinkage and (c) chemical reaction with the rubber. The net effect is an increase in volume, which is called “swelling”. The gain in weight is usually determined. The extent of swelling depends on temperature, time chemical nature and degree of cross linking of the polymer, loading level of filler and the relative molecular mass of both polymer and liquid, the hardness are usually determined after immersion and again after drying out the immersion liquid, the swelling ratio Q is defined as [31,33,34,35]

$$Q\% = (w_s - w_i / w_i) * 100\% \quad \dots\dots\dots(2.9)$$

The volume-swelling ratio r_v obtain by equation [19]

$$r_v = \frac{w_s - w_i}{w_i} \frac{\rho_p}{\rho_t} \times 100 \quad \dots\dots\dots (2.10)$$

Where, ρ_p is mass density of sample, and ρ_t is mass density of solvent, w_s is the weight of swollen sample, w_i is the initial weight of the sample. r_v Volume swelling ratio[36].

A decorative border with a gold-colored frame and floral motifs in purple, yellow, and green at the corners.

Chapter Three

MATERIALS

AND

EXPERIMENTAL

TECHNIQUES

3.1 Introduction

In this chapter, discuss the formation of rubber overlay kneading the base material in it (NR, NBR) supported with egg shell powder material (ESP) and carbon black (CB) (20 pphr) for the purpose of studying the damping properties of this superpose and its use in anti-ship vessels for the purpose of damping to reduce the resulting friction as a result of collision Ships in the anchorage. To study these properties, several tests must be conducted on the proposed pastries, as they are prepared under different prohibiting conditions, and the basis variable in which the proportions of the components reach to the dough appropriate for previous applications and then conform to international specifications. Where this chapter includes the presentation of the materials used in the formation of rubber dough and the method of its formation, as well as techniques and methods for testing the properties of rubber.

3.2 Materials

3.2.1NR used in this study is SMR 20 which has a specific gravity 0.92 gm /cm^3 , supplied by the Perlis, Malaysia.

3.2.2NBR (1052, acrylonitrile content 33%) was purchased from Nantex Industry Co., Ltd., China.

3.2.3Carbon black (CB) N375 supplied by Doudah, Iran. It is examined in accordance with DBP absorption (ASTM D136) and Iodine absorption (ASTM D135).

3.3 Eggshell powder (ESP), the eggshell wastes collected locally were washed thoroughly with water several times and dry in the sun for many days and crushed into smaller pieces. The pieces of eggshell wastes were then stirred mechanically to separate the eggshell and its membranes. The membranes were removed and the eggshell pieces were dried in the furnace for 1 hours at 100°C . After the drying process, the eggshell pieces were grounded into powder form using ultra centrifugal mill. The eggshell powder were placed in a box furnace for 2 hours at 400°C. Heat treatment was done onto the (ESP) to enhance the properties of the eggshell powder. Fig (3.1, a,b) show the eggshell powder (ESP) before and after heat treatment respectively.



Figure (3.1) a-before heat treatment.

Zinc oxide (97%) and stearic acid (99.4%) were supplied by Acidchem-International CO. Malaysia. 6PPD N- (1, 3 – Dimethyl butyl) – N – Phenyl – Para – Phenylenediamine (98%) was supplied by Shenyang Sunny joint Chemicals CO. China. MBS N- oxydi ethylene benzothiazole 2- sulfonamide (98.2%) supplied by ITT, India. The South Patrol Company supplied Paraphenic wax, processing oil. Sulfur was supplied by Al-Meshrak CO. Iraq.

3.4 Matrix batch

In this batches, natural rubber (NR) was used as a base material by (50 PPhr) with synthetic rubber Acrylonitrile Butadiene Rubber (NBR) and vulcanization materials , which included (zinc Oxide) and (Stearic Acid) as a stimulant , was used as material (MBS) as an accelerating substance, and (sulfur) as vulcanized material and (Process Oil) as a laxative, and table (3.1) shows the basic ingredients of the dough without additions:

(Table 3.1) Basic dough components without addition

Compounding ingredients	PPhr
NR	50
NBR	50
Stearic Acid	2
Zinc Oxide	3
MBS	1
Sulfur	2
6 PPD	1
Process oil	3
Egg shell powder	0,10,30,50

The dough was reinforced with egg shell powder material (ESP) and with different loading ratios to obtain rubber overlays (50NBR/50NR/ ESP) as in group (A) table (3.2) , as well as adding the same loading ratios in the presence of carbon black (CB-20 PPhr) to obtain rubber overlays (50NBR/50NR/ESP /CB) in group(B) as in table (3.3) .

Table (3.2) Ingredients of group (A)

Materials	A1	A2	A3	A4
NR	50	50	50	50
NBR	50	50	50	50
ZnO	3	3	3	3
Stearic acid	2	2	2	2
MBS	1	1	1	1
6 PPD	1	1	1	1
Carbon black	0	0	0	0
Process Oil	3	3	3	3
Egg Shell powder	0	10	30	50
Sulfur	2	2	2	2

Table (3.3) Ingredients of group (B)

Materials	A1	A2	A3	A4
NR	50	50	50	50
NBR	50	50	50	50
ZnO	3	3	3	3
Stearic acid	2	2	2	2
MBS	1	1	1	1
6 PPD	1	1	1	1
Carbon black	20	20	20	20
Process Oil	3	3	3	3
Egg Shell powder	0	10	30	50
Sulfur	2	2	2	2

3.5 Equipment and Instruments

3.5.1 Laboratory mill

A two-roll mill was used as shown in Fig. (3.2), it consists of two hollow cast iron rolls of cylindrical shape of 150mm in diameter and 300mm in length, having provision for passing cold water or steam through the rolls. The mixing of powder takes place at the compressive zone of the roll nip, where the mixing take place along the circumferential direction of the rubber bank on the mill. In mill mixing [8], temperature control is very important so, chilled water is passed through the rolls at a regulated flow rate to remove excessive heat developed during mixing, and prevents scorching of compound, and heat is also necessary in some processes to warm up the rolls instead of cooling them.



Figure (3.2) Laboratory mill.

3.5.2 Hydraulic press

The vulcanization processes are function of pressure, heat, and time according to the specification test. The preparation processes of samples were carried out by the hydraulic press has maximum pressure equal to 700bar, the hydraulic press is equipped with thermocouple and maximum temperature is equal to 300°C, see Fig. (3.3) .



Figure (3.3) Hydraulic press

3.5.3 Mould for Testing hardness, and rebound resilience.

For preparing samples of hardness, rebound resilience and density tests, the mould of the laboratories of Tires Company was used. The mould consists of three parts , the middle part has dimension of (200 * 180 * 4) mm which contains nine circular equivolume cavity with (40 mm) in diameter and 4 mm thickness, while one of other two parts is the bottom base and the other is a cover , they have a dimension of (200 * 180 * 10) mm as shown in Fig. (3.4) .

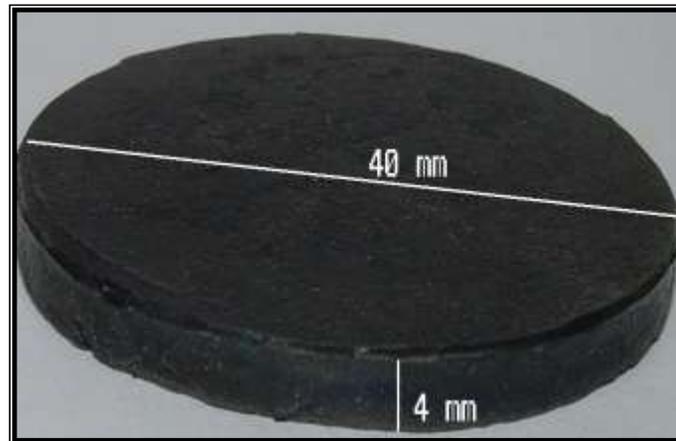


Figure(3.4) Mould of samples for testing hardness, rebound resilience and specific gravity.

3.5.4 Preparation of Samples for Hardness (shore A), Rebound resilience and specific gravity tests

Tests were carried out for all samples according to the following steps:

1. Mould in Fig. (3.4) is heated to 100 °C.
2. Removed the mould from the oven with protective gloves and lubricated all its sides.
3. After preparation of the mould, an appropriate quantity of recipe was put in the mould.
- 4- The mould was put in the hydraulic press under 20MPa and at temperature of 160 °C for 20 min for performing vulcanization process figure (3.3).
- 5- The samples were removed from the mould and left for 10 – 15 hours to cool at room temperature before test.
- 6-The hardness sample, rebound resilience and specific gravity test sample is shown in Fig. (3.5).



Figure(3.5) The samples for hardness, rebound resilience and specific gravity tests.

3.6 Equipment for Rebound Resilience Measurement

The Dunlop trips meter takes the form of an out of balance metal disc revolving on virtually frictionless bearing and which acts as a slow moving pendulum. The test piece is placed against a rigid support so that it receives a blow at its center. A circular scale is provided for measuring the angle of displacement .The measured quantity is the angle to which the disk rebounds after the hammer strikes the specimen. Tests are carried out according to ASTM D1054 as shown in Fig. (3.6).



Figure.(3.6): Equipment for resilience

3.7 Equipment for Hardness (shore A) measurement

The International Hardness test is used in the measurement of the penetration of rigid ball (according to Brinall method) into the rubber specimen. The diameter of the ball equal to 2.5 mm; sample for hardness prepared in section (3.7), under specified conditions. the equipment shown and using a device (Shore A) Fig.(3.7). The scale of degrees is so chosen that zero represents a material having elastic modulus equal to zero and 100 represents a material of infinite elastic modulus. The scale covers all the normal range of hardness. The tests were carried out according to ASTM D 1415 specifications .



Fig.(3.7) Equipment of hardness

3.8 preparation of campsites Rubber (50NBR/50NR / ESP)

and (50NBR/50NR / ESP / C.B- 20 pphr) :

1. The rubber piece is passed (50NR) by weight (200pphr) and (50NBR) by weight (200pphr) of the base dough through the two rolls and several times from the rolling mill and by reducing the distance between the two rolls so that the rubber piece is homogeneous and the process is done at a temperature of 45C⁰ .

-
2. Add a substance that activates the accelerating substance (stearic acid) and by continuous mixing to obtain a homogeneous mixture for 5 minutes.
 3. (Zinc oxide) is added, which is an activated substance for the accelerator, by continuous mixing with rubber and passing it several times during the two rolls until the homogenization takes 5 minutes as well.
 4. Add the precipitated substance (MBS) to the rubber to reduce the time of vulcanization and increase the percentage of vulcanization with continuous passage within the two rolls and for a period of 5 minutes.
 5. Add drops of oil during the mixing process to soften the rubber for homogenization.
 6. Add the sulfur as vulcanizing material and also with continuous mixing for 5 minutes.
 7. Add the egg shell powder with weight proportions (0, ,10, 30,50pphr) to group A , while group B has carbon black and the same weight proportions to the egg shell powder.
 8. The dough is wrapped around the two rolls for the purpose of final homogenization, with the distance between the two rolls reduced to (2mm) , then cooled and the dough left for a period of (24 hr), then placed in special molds to prohibit the examination samples.

3.9 .1 Measuring the weight of samples.

Using a device (Sensitive electronic balance) we calculate the weights of the samples in the form of two groups, group A, which contains egg shell powder in different proportions and there is no carbon black , group B also contains egg shell

powder in different proportions as well as carbon black , using a device (Sensitive electronic balance) figure (3.8). It is one of the necessary devices that are used in laboratories , in order to determine the weights of the required quantities , and when using it is taken into account that it is clean from inside and outside, and it is preferable to use the same balance throughout the period of preparing experiments to avoid error and this type of scales has the ability to determine weights with high accuracy (0.001-g accuracy \pm) according to For international specifications (ASTM D3182) (Weighing of Materials procedure) .



figure (3.8):- Sensitive electronic balance

The results of the weights for group A and group B were obtained, according to tables (3.4) and (3.5) .

This device was worked on in the laboratory of the College of Education for Pure Sciences / Department of Physics / University of Babylon.

Tables (3.4) Ingredients of group A

Egg Shell Powder	Weight (gm) Before swelling	Weight (gm) After swelling
0	6.011	6.0325
10	6.264	6.2802
30	6.947	6.9795
50	7.214	7.2326

Tables (3.5) Ingredients of group B

Eggshell Powder	Carbon black C.B	Weight (gm) Before swelling	Weight (gm) After swelling
0	20	6.441	6.4590
10	20	6.815	6.8334
30	20	7.367	7.3837
50	20	7.158	7.1723

3.9.2 Rebound Resilience test method

The Dunlop trips meter was used to measure the rebound resilience show Fig (3.7) and the specimen show Figure (3.6) so the testes was carried out according these steps:

1. Put the samples in oven 50° C at the interval time one hour.
2. Put the sample in the suitable place in the trips meter.
3. Allow to the pendulum to strike the specimen.

-
4. Recording the rebound resilience R after the hammer strikes the specimen.
 5. Recording the damping time T after the hammer stopped.
 6. Recording the angle of incidence for each stroke of the pendulum.

3.9.3 Hardness test method:

Hardness tests were carried out by hardness equipment according to International Rubber Hardness Degrees (shore A), the samples are explained in section (3.5) The sample are 40 mm diameter and 4mm thickness as shown in Figure (3.6). The test temperature is 25°C. The test procedure was as in the following steps:

- 1- Dusting of the sample surface was slighted with tack.
- 2- The Vibrator was started.
- 3- The specimen was supported on a horizontal rigid surface.
- 4- The foot was lowered to rest on the plain surface of tests specimen.
- 5- The plunger was pressed with minor load on indenter for 5 seconds.
- 6- The pointer was adjusting to read 100.
- 7- The plunger was pressed to add the major load on the indenter within 30 seconds.
- 8- The reading was recorded on the gauge as (shore A),

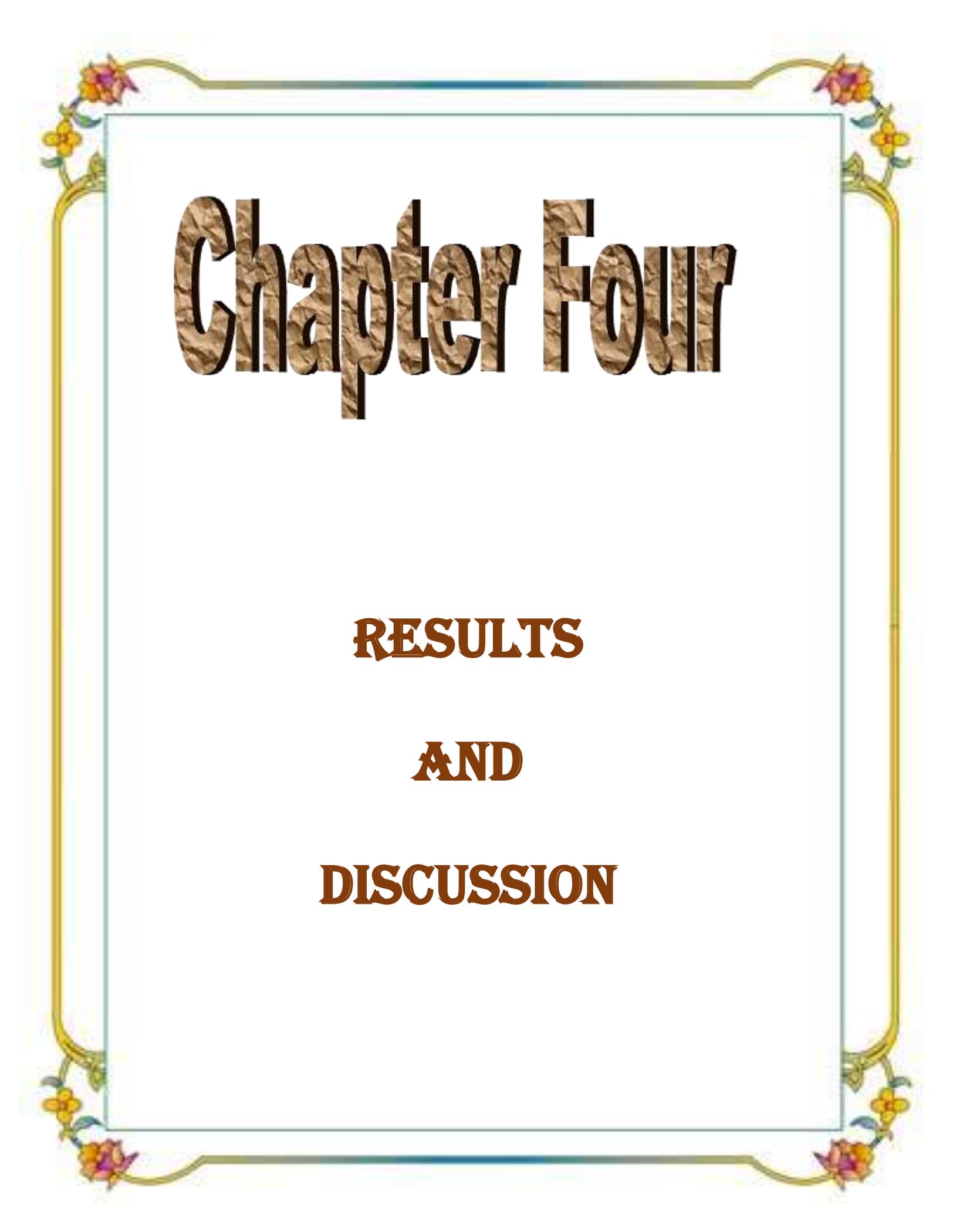
3.10 Swelling Test

The samples for the best recipes were prepared according ASTM D471 and carried out tests in saline water (30 gm for 100ml) were reinforced with 20 pphr of carbon black and (0,10,30,50)pphr of reclaim or waste having the same hardness samples dimensions.

These samples were immersed in the container, which contained saline water at 25^o C.

Dry weight of the samples had been taken before immersion, then the weight had been recorded after immersion in saline water through 96 hour. The mass of the sample measured by electronic digital balance with 0.0001 gm accuracy, the swelling was calculated according the equation.

$$Q\% = (w_s - w_i / w_i) * 100\%$$



Chapter Four

RESULTS

AND

DISCUSSION

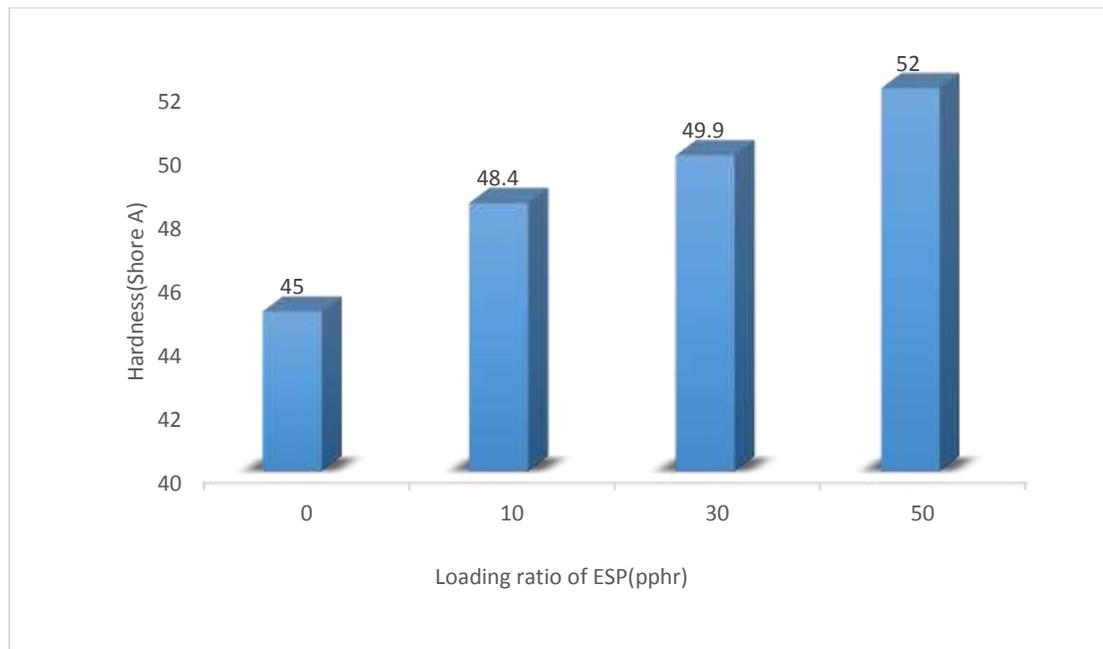
4.1 Introduction

This chapter presents all results obtained and study properties (Hardness, Rebound Resilience, Damping Time, swelling ratio). This chapter includes practical results obtained from Measurements and checks made on rubber overlays. Based on these results, the possibility of using eggs shell powder in many applications was discussed, where two groups were prepared (50NBR/50NR/ ESP) It contains eggs shell powder with loading ratios (0, 10, 30, 50 pphr) ((group A)). While the second composite (50NBR/50NR/ESP/C.B 20) for the same loading ratios in the presence of carbon black represent (group B).

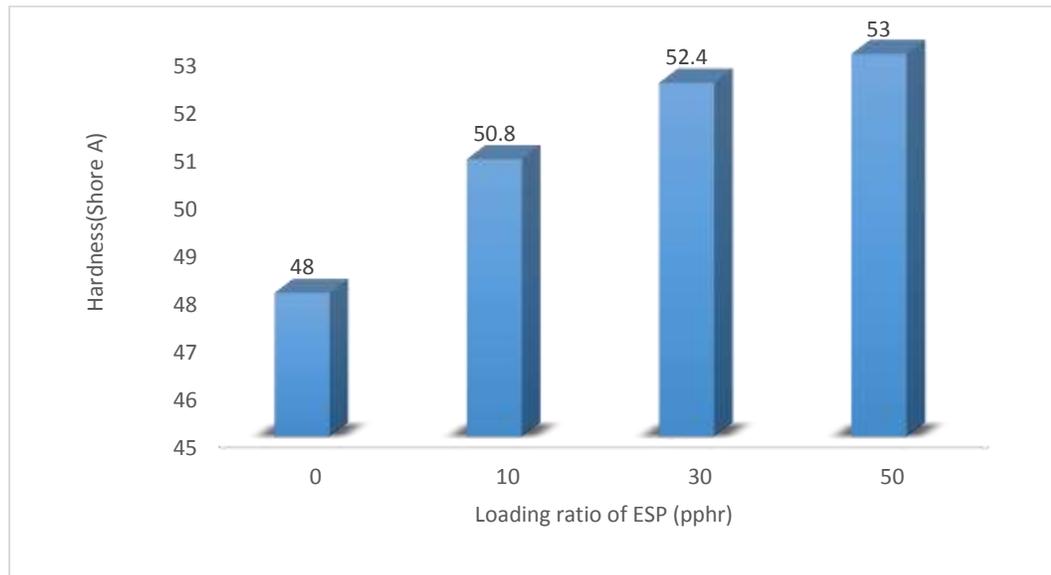
4.2 Hardness of (50NBR/50NR/ESP) and (50NBR/50NR/ESP/C.B-20pphr) Composites.

The hardness of two groups were examined by shore A, these results are shown in figures (4.1) and (4.2), it indicate that the addition of the (ESP and C.B- 20pphr) filler showed a marked Increase in hardness, this result is expected because of more fillers incorporated in the rubber matrix. It is well known that the addition of the Filler in rubber compounding leads to a linear increase in materials Hardness, moreover, this attributed to due to the presence of carbon black Resulted increasing number of high active sites leads to active Crosslinking sites between

the rubber chains and rubber-C.B -20pphr Rubber- ESP interaction and filling the vacuums between the rubbers Chains this behaviour is agree with the results of [8,18,37]but with Other Fillers.



Figures (4.1) Relation between Loading ratio (ESP) and hardness (50NBR/50NR/ESP) without CB group A

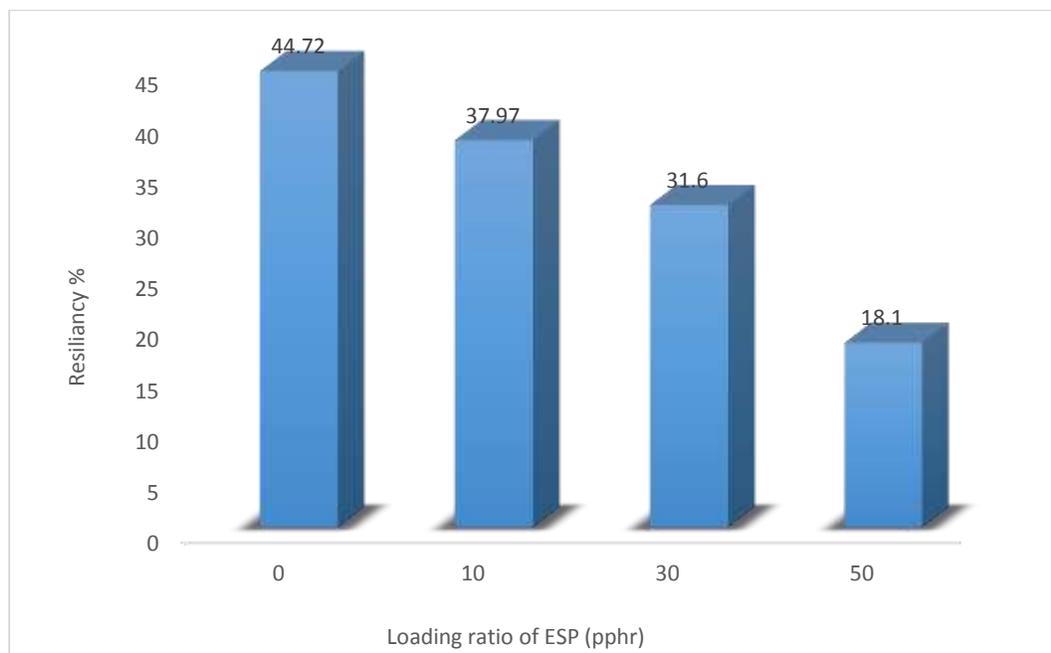


Figures (4.2) Relation between loading ratio (ESP) and hardness (50NBR/50NR/ESP- with CB20) group B

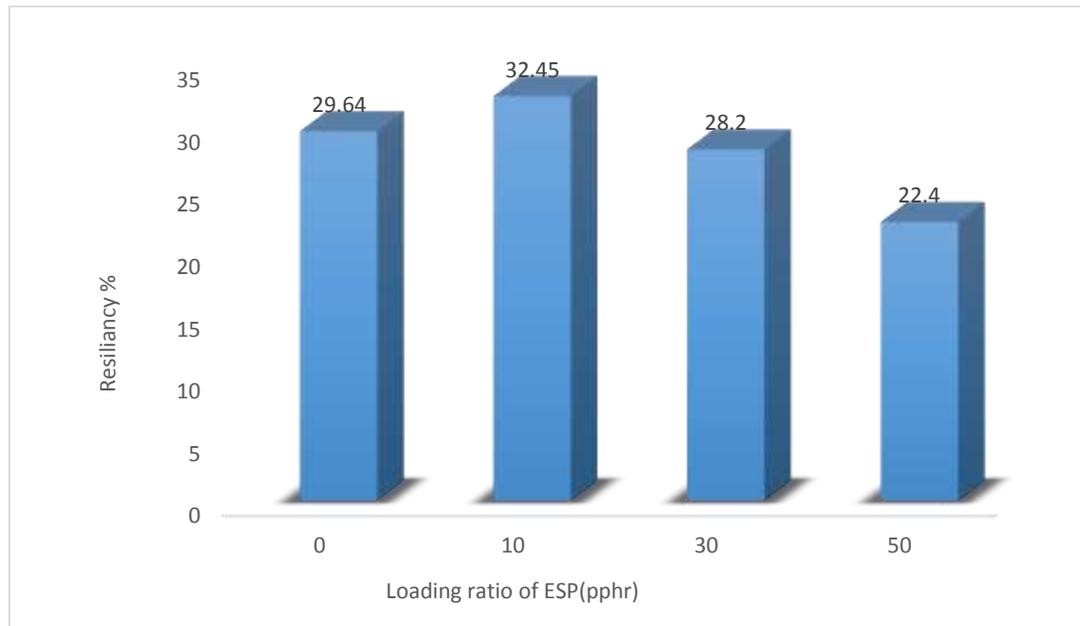
4.3 Rebound Resilience of (50NBR/50NR / ESP) and (50NBR/50NR / C.B – 20pphr / ESP) composites.

The rebound resilience of the two groups was investigated by Dunlop trips meter equipment and the results are shown in the figures (4.3) and (4.4). These Figures indicated that the relation between the rebound resilience and the loading percent of C.B – 20pphr and ESP, while the rebound resilience decreasing in linear behaviour as the mixed of C.B -20 pphr and ESP. The decrement in the rebound resilience is due to the excess of the loading level of these mixes in composites and increasing physical cross-linking between rubber blends and C.B-20 pphr, ESP this leads to filling the vacuum between the rubber chains, so it is inversely

proportional with increase damping and Hardness. This property is very important for the dampers due to the ability of absorbent to the sudden shocks .This absorption caused by high strain elastic deformation, which results in dissipation of the impact energy without fracture or fatigue failure. This behaviour is agreement with [21,29]



Figures (4.3) Relation between the rate of loading (ESP) and rebound resilience (50NBR/50NR/ESP) without CB group A

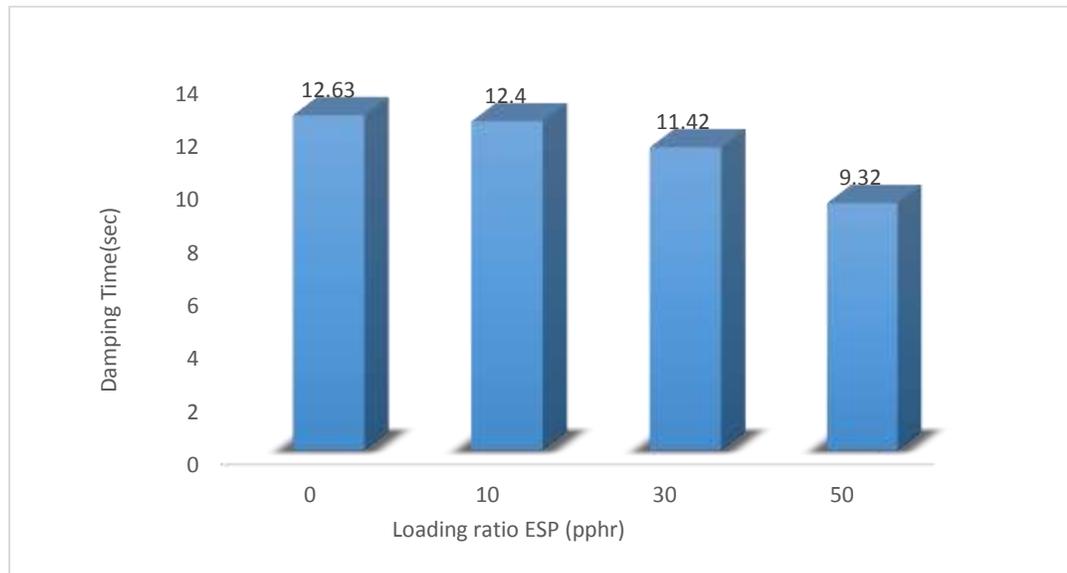


Figures (4.4) Relation between the rate of loading (ESP) and rebound resiliency (50NBR/50NR/ESP-20 CB) group B

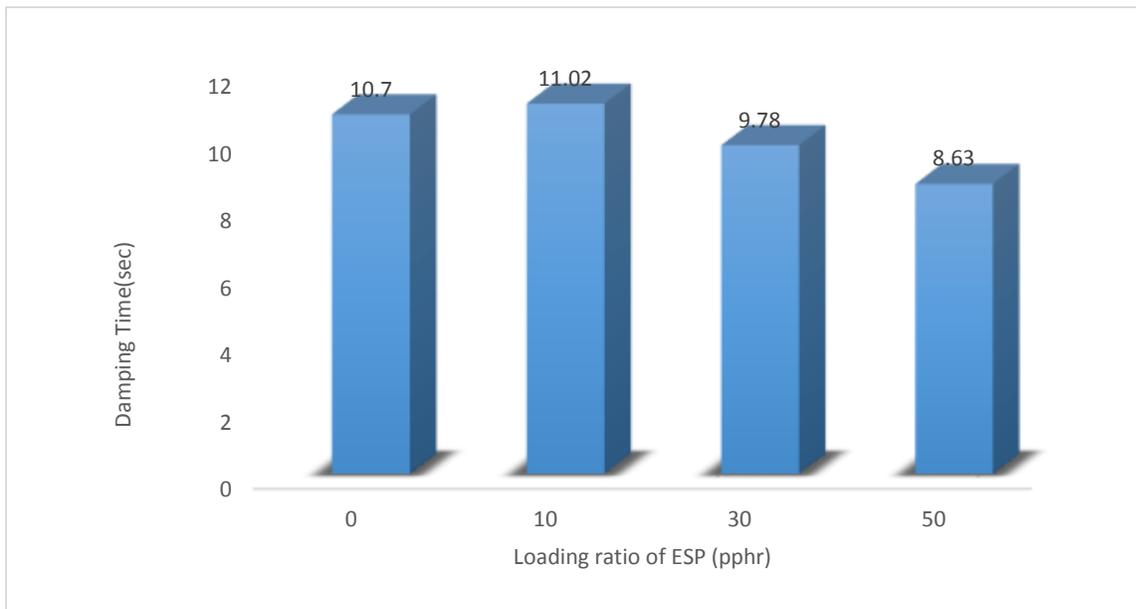
4.4 Damping Time of (50NBR/50NR / ESP) and (50NBR/50NR / C.B -20pphr / ESP) composites.

The damping time of two groups were examined by Dunlop trips meter equipment, and the results are shown in the Figures (4.5) and (5.6). These Figures indicate that damping time decreased with increasing the loading level of ESP and 20CB. This decrement attributed to the same causes of resilience decrement. In addition increased loading level of C.B-20pphr and ESP to increase the hysteresis and growth of small physical and chemical attractive ranges, and the molecular

interaction between rubber chains and Carbon black grain surfaces since it have higher polarity and interactive site. This behaviour is agreement with [23]

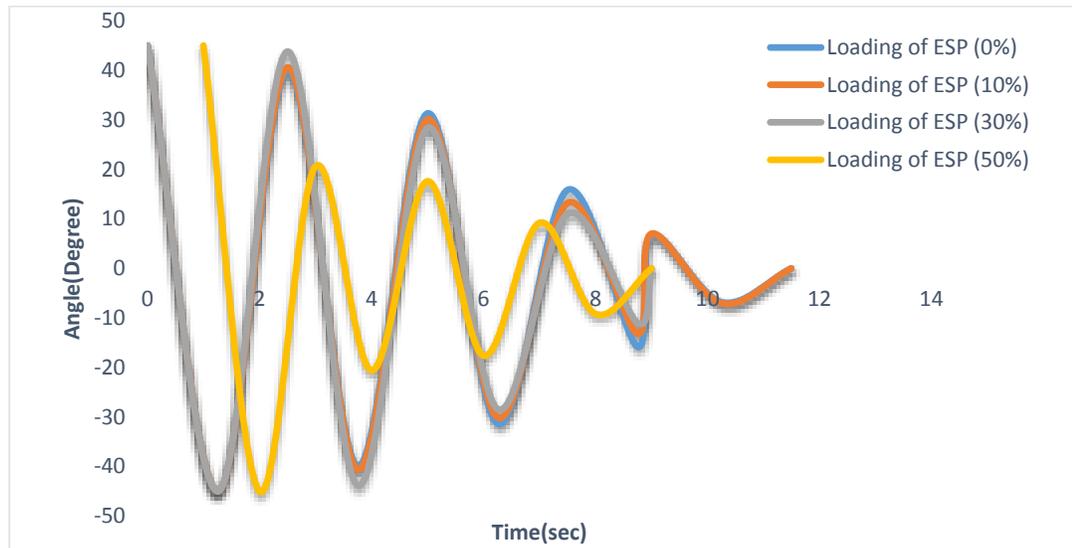


Figures (4.5) Relation between the loading ratio (ESP) and the damping time (50NBR/50NR/ESP) without C.B20 group A

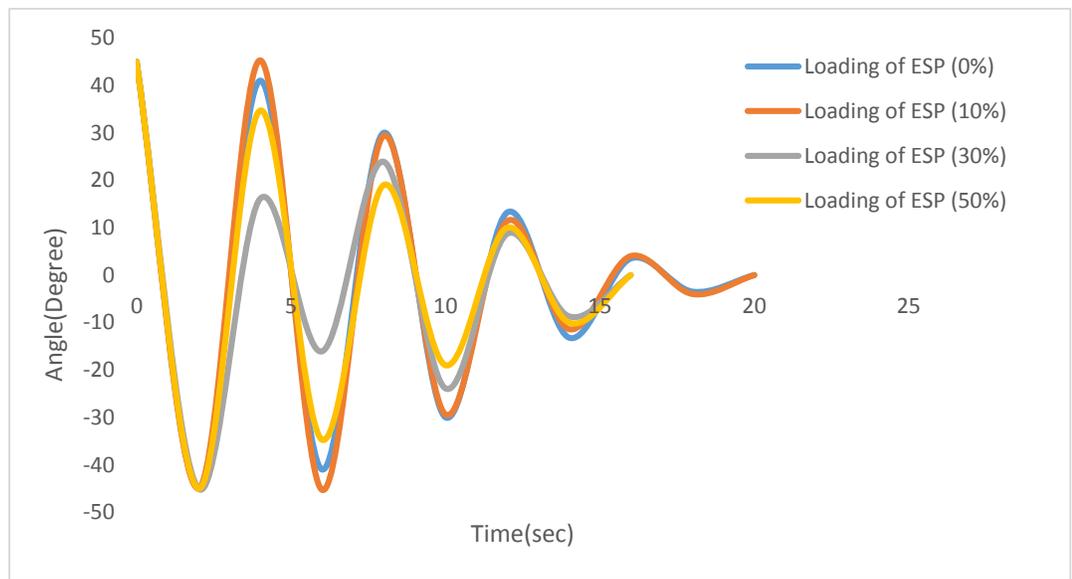


Figures (4.6) Relation between the loading ratio (ESP) and the damping time (50NBR/50NR/ESP) with C.B20 group B

A scale was provided to measure the angle of displacement, and the measured quantity is the angle to which the disc bounces after the hammer hits the sample. The tests were taken with a Dunlop trip counter with a rotating metal disc and were implemented according to the specifications (ASTM D1054). The results showed a significant decrease in the angle of elasticity, and this result is expected due to the increase in the ratios (ESP) of the compound. Increasing the filler in the rubber overlays leads to a decrease in its flexibility and thus a decrease in the angle. This decrease is more noticeable in group (B) due to the presence of carbon black, the results are shown in the Figures (4.7) and (4.8).



Figures (4.7) Relation between the resilience angle and damping time for (50NBR/50NR/ESP) without CB group A



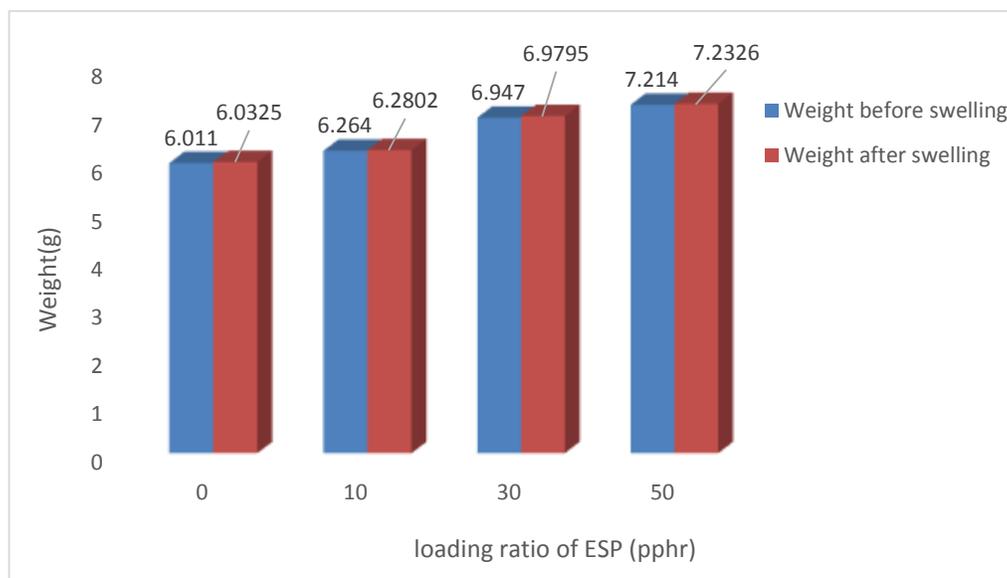
Figures (4.8) Relation between the resilience angle and damping time for (50NBR/50NR/ESP) with CB group B

4.5 Swelling ratios:

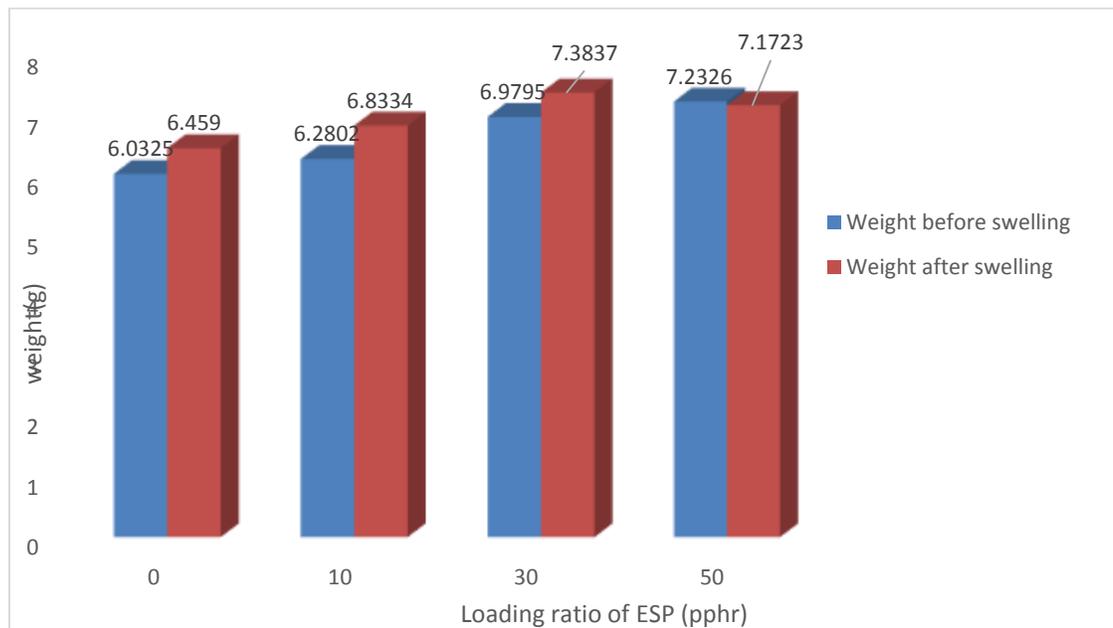
The swelling ratios of the rubber compounds in saline water was investigated through the results which had been, measured by weight specimens before and after immersion (96 hour) as shown in Figure (4.9)-(4.14).

These results indicated that no variations in these values swelling of small deviation after immersed in saline water. Therefore, the ratio of swelling was very small for all these tested rubber composites. In addition this Figure indicated that the swelling ratio increase with increase loading level of ESP because of the swelling ratio is depend on type of elastomers ,cross-linked density, type of filler and loading level of filler.[35]

From the figure (4.9) and (4.10) that the weight of the samples has increased by very few percentages, and these percentages increase as the loading of (ESP) increases in the group (A) which does not contain carbon black. As for the group (B) be just the opposite, where a decrease in the change in weight of the samples as the loading (ESP) increases and the reason is due to the presence of carbon black.

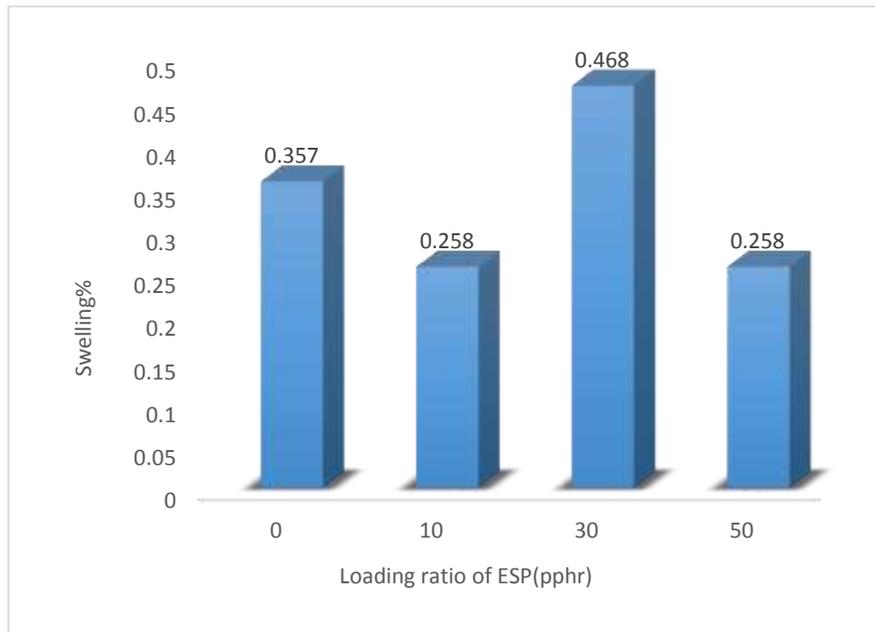


Figures (4.9) Relation between the loading ratio (ESP) and the weight for (50NBR/50NR/ESP) without C.B20 group A

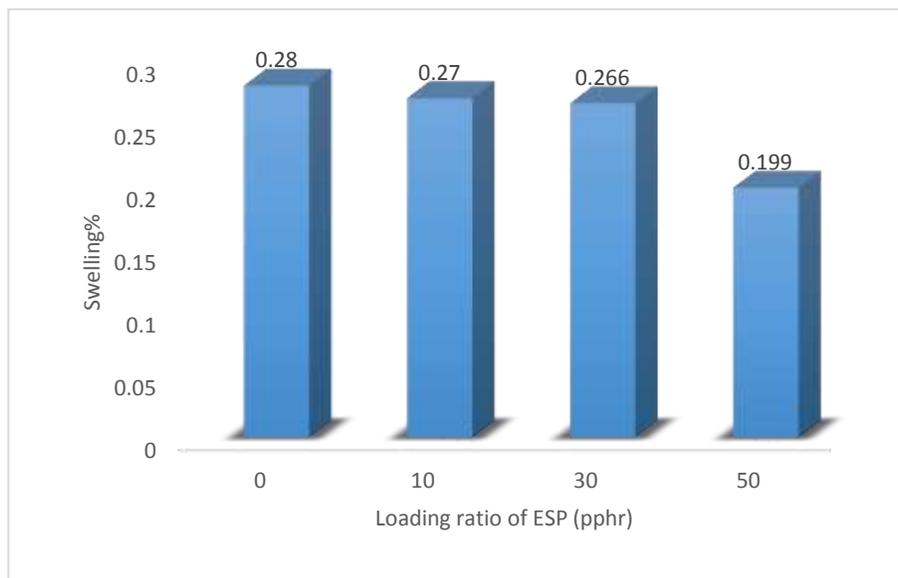


Figures (4.10) Relation between the loading ratio (ESP) and the weight for (50NBR/50NR/ESP) with C.B20 group B

The swelling ratios of the rubber compounds in saline water was investigated through the results which had been, measured by weight specimens before and after immersion , these results indicated that no variations in these values swelling of small deviation after immersed in saline water. Therefore, the ratio of swelling was very small for all these tested rubber composites. In addition this Figure indicated that the swelling ratio increase with increase loading level of ESP because of the swelling ratio is depend on type of elastomers ,cross-linked density, as shown in Figure (4.11) and (4.12).

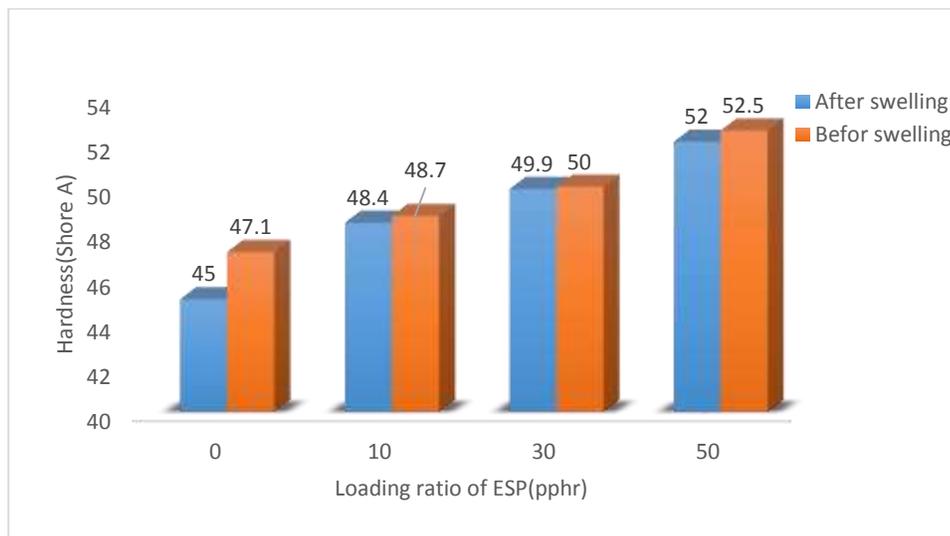


Figures (4.11) Relation between the loading ratio (ESP) and the swelling for (50NBR/50NR/ESP) without C.B20 group A

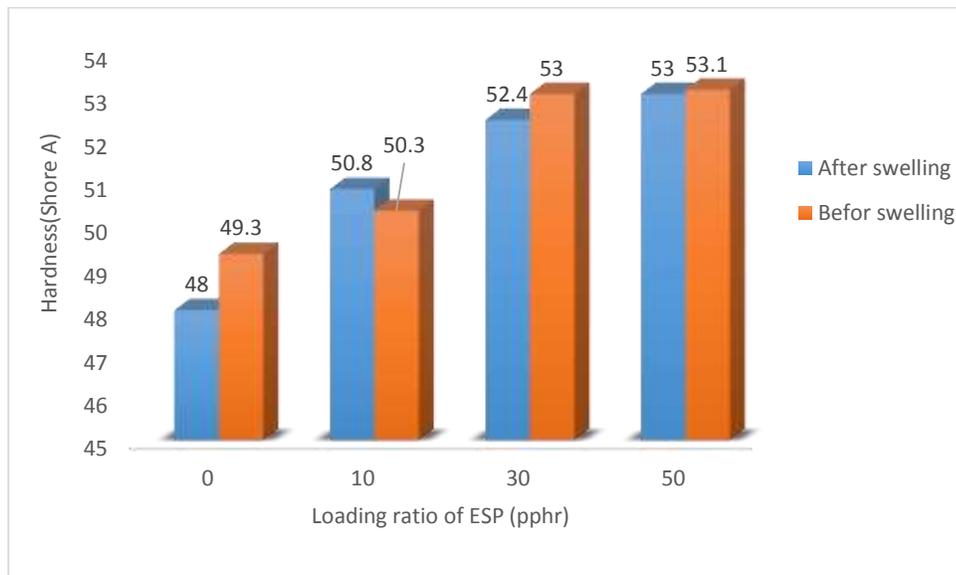


Figures (4.12) Relation between the loading ratio (ESP) and the swelling for (50NBR/50NR/ESP) with C.B20 group B

The hardness test was conducted before and after immersing the samples in salt water, and by viewing the figure (4.13) and (4.14) a slight increase in the hardness of the samples after immersion in the solution, and it decreases with increasing the loading percentage (ESP). The reason for this is that the rubber components do not respond to the solution, and these changes are reduced in the group (B) due to the presence of carbon black.



Figures (4.13) Relation between Loading ratio (ESP) and hardness after and before swelling (50NBR/50NR/ESP) without CB group A



Figures (4.14) Relation between loading ratio (ESP) and hardness after and before swelling (50NBR/50NR/ESP) with CB group B

4.6 Conclusion

1- There is a change in the hardness of the rubber composites (50NBR/50NR/ESP) and (50NBR/50NR/ESP), as the hardness increases with the increase in the loading ratio of the egg shell powder (ESP) to the composite with carbon black (CB 20 pphr) and without (CB 20 pphr). This is due to the filling of the volume between the rubber series by (ESP) and (CB).

2-Damping time of Rubber decreases as the percentage of eggshell powder loading of the composites increases, this leads to use it in Damping application.

3-The swelling ratios of the rubber composites decreased when the eggshell powder (ESP) was increased to Rubber with carbon black and without (CB 20 pphr), and the swelling ratio be less in (50NBR/50NR/ESP-CB) Composite.

4.7 Future Works:

1-Study of the mechanical Properties of (NR 40/NBR60 / ESP) and (NR 40/NBR60/ C.B -50pphr / ESP)

2- Study of the thermal properties of (NR 50/NBR50 / ESP) and (NR 50/NBR50 / C.B -20pphr / ESP).

3- Study of the damping properties of a composite (NR 30/NBR50 / ESP/CB40 pphr)

4 - Study of the damping properties and structured of a composite (NR 50/SBR50 / ESP/CB40 pphr)

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الخلاصة

تم في هذا البحث تداخل دفعات مطاطية مركبة مكونة من المطاط الطبيعي (50NR) ومطاط اكريلونتريل بوتادين (50NBR) كمادة اساسية وتم اضافة مسحوق قشور البيض (ESP) وبحجم حبيبي اقل من (222 MM) وبسبب تحميل (0,10,30,50 pphr) ايضا مادة اسود الكربون (CB) كمادة داعمة وبنسبة تحميل (20 pphr) حيث تم الحصول على مجموعتين من المركبات : المجموعة (A) تتكون من (50NBR/50NR/ESP) المجموعة (B) وتتكون من (50NBR/50NR/ESP/CB-20pphr) تمت دراسة بعض الخواص الميكانيكية مثل الصلابة حيث تم قياسها باستخدام جهاز (shore A) وحسب المواصفات (ASTM D 1415) ونسبة الانتفاخ لهذه المركبات المطاطية حسب المواصفات (ASTM D 471-98) كذلك خواص التخميد مثل مرونة الارتداد وزمن التخميد باستخدام جهاز

(Trips meter) أظهرت النتائج زيادة في قيم الصلابة مع زيادة في نسبة التحميل وللمجموعتين (A و B) والزيادة أكثر للمجموعة (B) . كما أظهرت النتائج ان هناك انخفاض واضح في قيم مرونة الارتداد وزمن التخميد مع زيادة نسبة تحميل (ESP) لكلا المجموعتين , ويزداد هذا الانخفاض بالنسبة للمجموعة (B) . وفيما يتعلق بالانتفاخ بالمياه المالحة فقد وجد ان هذه التراكبات لها زيادة طفيفة عند الغمر في المياه المالحة



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لمتراكبات نانوية (خليط مطاطي/اسود الكربون)

بحث مقدم

إلى مجلس كلية التربية للعلوم الصرفة / جامعة بابل كجزء من متطلبات
نيل درجة

الدبلوم العالي تربية / فيزياء المواد وتطبيقاتها

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