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***Research paper***

# Optimization of Manufacturing Parameters Affecting on Characterization of Porous Sintered Tin-Bronze Alloy

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## Abstract

Porous sintered bronze alloy has widespread applications in various engineering fields such as filtration systems, self-lubricating bearings, fluid flow control, heat exchanger ..etc. In this paper, powder metallurgy technique using NaCl particles high purity (99.5%) as a space holder (pore-forming agent) was employed to produce a rigid porous structure . The paper aimed at studying and identifying the effect of manufacturing parameters (NaCl wt. %, compacting pressure, sintering temperature and sintering duration) on the characteristics of porous tin-bronze alloy . According to Response Surface Methodology analysis a multi-optimization method based on desirability function used to obtain the optimum process conditions that can be used in manufacturing of porous sintered tin-bronze structure. These optimum conditions were: (35.36 wt.%NaCl), (40 MPa) compacting pressure , sintering temperature of (193.5 $^{o}$C) and sintering time of (180 min) to get maximum porosity of (66.32 %) , permeability coefficient of (2.96*10$^{-3}$ cm/min.) and micro-hardness of (57.12 Hv).

***Keywords:*** *Porous sintered bronze alloy; powder metallurgy; porosity; permeability coefficient; RSM.*

## 1. Introductions

Porous metals have intentionally created pores in their structure to give them a very advantageous combination of physical and mechanical properties .Such materials have very low specific weight , good stiffness-to-weight ratio, larger capacity of damping , ability of sound-absorbing , high permeability combined with high thermal conductivity[1].These unique engineered materials provide specialized products for many applications like filtration, fluid flow control, self-lubricating bearings, medicine (bone and dental implants) and battery electrodes [2]. Materials can be selected from a wide variety of powdered metal depending on the combination of application requirements and economics. The most common porous powder metallurgy P/M materials are bronze, stainless steel, nickel and nickel based alloys. Other materials such as titanium, aluminum, copper, platinum, gold, silver, niobium, tantalum and zirconium are also fabricated into porous metals from powder [3]. The characteristics of a porous material depend on the size, arrangement and shape of the pores, the total pores volume relative to the volume of the material, and composition of the material itself. The unique combination of physical and mechanical properties offered by porous metals, that cannot be obtained with dense metals, or either dense or porous polymers and ceramics, makes them advance materials [4].A major classification of porous metals are open-cell, close-cell, or a combination of the two types. The size, shape and location of pores within the matrix are varying , depending on the parameters of the fabrication process [5].Porous P/M materials when special characteristics are required such as good mechanical properties, rigidity, corrosion resistance, uniform porosity and controlled permeability. Bronze powders consisting of 90-92wt % copper and 8-10 wt % tin are the most common material for a porous bronze P/M components such as filters and self-lubricating bearings [6].

The presented paper focused on analyzing and optimizing the effect of manufacturing conditions on physical and mechanical properties of porous sintered tin-bronze alloy prepared by powder metallurgy technique. Mathematically models design to study influences of pore-forming agent (NaClwt. %), compacting pressure ,sintering temperature and sintering duration on the porosity, the coefficient of permeability and micro- hardness by use Responses Surfaces Methodology (RSM).

## 2. Experimental Details

### 2.1. Materials Used

Powders of copper, tin and NaClwere used in this study .The powders have the specifications demonstrated in Table 1.

**Table 1:** Purity & Average Particles Sizesof Used Powders.

| Powder | Purity | Average Particles Size (µm) |
|---|---|---|
| Cu | 99.83 | 30.62 |
| Sn | 99.78 | 23.27 |
| NaCl | 99.50 | 275.81 |

### 2.2. Fabrication of Porous Sintered Tin-Bronze Structure

Copper was mixed with 10 wt.% of tin for all samples in this study and then (20, 30, 40 wt.%) of NaCl powder was added as a pore forming agent . Wet mix used with 2%wt of acetone. The mixing processes achieved via steel balls mill, types: (STGQME-15⁄-2) for 2 hours in order to get the perfect and homogenous distribution of powder particles. The mixture was

dried at 60 $^{O}$C for 30 min. the compacting by doubles actions steel dies carried-out in electrohydraulic compacting machine types: (CT440-CT430,USA).A constant loading rate of 0.3 KN/sec was used for all compacting processes with a duration of 8 min for the achieved pressure. Two type of samples prepared, as the fallowing : disk samples with (12.8 mm) diameters and (3 mm) heights using for density, porosity, harnesses', permeability tests and cylindrical samples with (12.8 mm) at diameters and (8 mm) at heightsusing to microstructure and X-Ray diffraction tests. A compacting pressure of (40, 60 and 80)MPa was used to prepare the samples. The sintering process done by vacuums high-temperatures tubes furnace through pressures of $10^{-4}$ tor. A sintering-program illustrated in Fig.1was used. The samples left inside the furnaces to cools down. The sintered samples were immersed in a distilled water via electro-magnetic stirrer type:( F91T), for 8 hr at 100 $^{0}$C to remove and dissolve NaCl. Fig. 2 shows some samples prepared after this process.

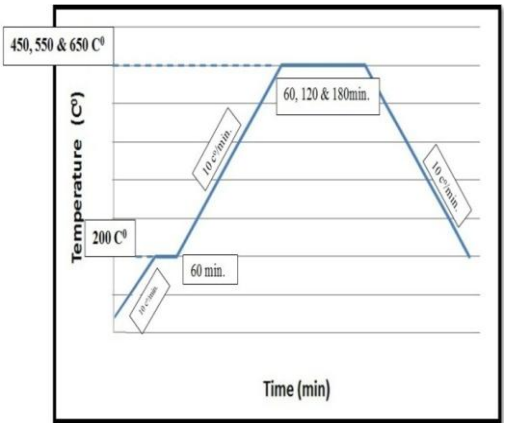



**Fig. 1:** The Sintering Program of the Green Samples

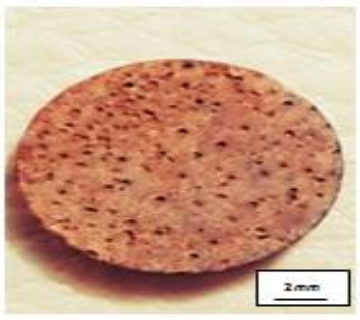


**Fig. 2:** The samples after removing NaCl

### 2.3. Microscope & X-Ray Diffraction Tests

Optically microscopes analyses and XRD analyses perform to prepared sintered samples for identify created phases. Optically microscopesanalysis carried-out to sintered tin-bronze alloys after polish and etch process, using Optically microscopestypes: (1270XEQMM310TUSBE). XRD analyses covering the samples via use XRD analyzers types: (MiniFlexe 20) with test situations of: (Targets: Cu, wave lengths of 1.54050A°, voltages and currents are 30 KV and 15 mA respectively, scanning speeds of 2deg/min., and scanning ranges of (20°-80°).

### 2.4. Mechanical and physical Tests

- **Porosity test**

Porosity test of the final porous sintered tin-bronze samples were measured according to ASTM B-328 asfollows [7][8]:

❖ After drying at 100 °C for 5 hours in vacuum furnace the sample was weighted and this weight is demoted by A.

❖ At room temperature, the sample is completely immersed in oil for 30 minuts .A suitable evacuating pump was used , to decrease the pressure.

❖ Weighting the fully impregnated sample in air, and the results demoted by B.

❖ Weighting the fully impregnated specimen in water, it is mass C.

porosity has been calculated by the following equations [7]:

$$P = [\frac{B-A}{D_o(B-C)} \times 100] D_W \quad (1)$$

Where: $D_o$ = density of oil(0.8 g/cm³).
$D_W$=density of water at 25 $^{0}$C.

- **Permeability Test**

The permeability of the porous sintered samples were measured by constant head method, [9]. This method is carried out by applying a constant head of water over the sample and collect the permeates through the pores of porous media. The permeability is calculated according to Darcy's equation (2) [9].

$$K = Q.T/A.h.t \quad (2)$$

where:
K= Permeability coefficient, cm/min.
Q= collected volume of a liquid, $cm^3$.
T= thickness of porous media (0.3cm) .
A= cross sectional area of porous media, ($1.287cm^{2)}$ .
h= constant head, (54)cm, and
t= time required to collect the volume of liquid, (5min) .

- **Hardness Test**

Vickers hardness test was carried out to the sintered samples by micro Vickers hardness device of type: (Digital Micro Vickers Hardness Tester TH 717) using a load of 300g for 10 sec with a square base diamond pyramid. The hardness' recording as an averages of 5 readings for everysample.

## 3. Results and Discussion

### 3.1. X-Ray Diffraction & Microscope Analysis

Fig.3 & 4 represent of (XRD) tests. Fig. 3 is the resulted (XRD) pattern for a sample prepared with 20 wt. % NaCl by using a compacting pressure of 40 MPa , a sintering temperature of 450 °C and a sintering time of 60 min. Fig.4 is the resulted (XRD) pattern for a sample prepared with 40 wt. % NaCl by using a compacting pressure of 80 MPa , a sintering temperature of 650 °Cand a sintering time of 180 min. The XRD results for both samples insure the presence two phases, α–phase as the matrix of the alloy while the second phase is ε – ($Cu_3$-Sn) as intermetallic phase.

The microstructures of tin-bronze alloy are shown in Fig. (5- a & b). Observed from images that the samples after sintering process have a microstructure composed of two regions, light (bright) which represents α-phase network matrix of the microstructure, and the other region is dark which refers to as fine ε-phase (intermetallic compound). The microstructure results are in good agreement with [10].

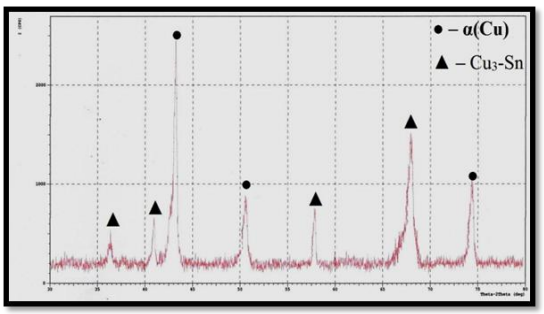



**Fig. 3:** XRD Pattern of Sample Prepared With 20 wt. % Nacl , 40 MPa Compact Pressure, 450 ºC Sintering Temperature and 60 Min. Sintering Time

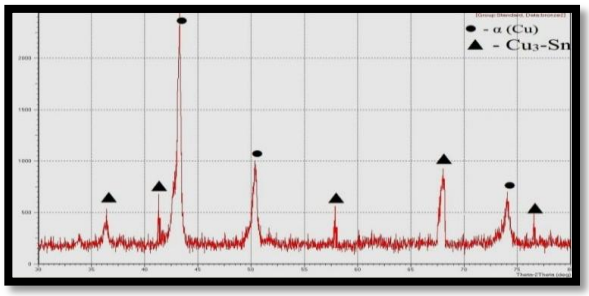



**Fig. 4:** XRD Pattern of Sample Prepared With 40 wt. % Nacl , 80 MPa Compact Pressure, 650 $^{o}$C Sintering Temperature and 180 Min. Sintering Time

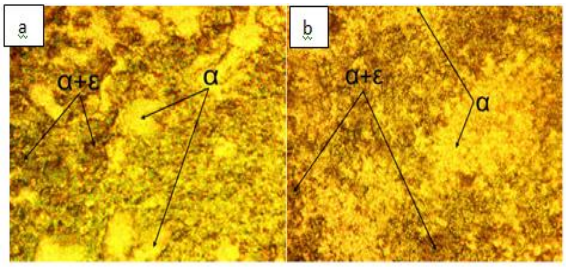



**Fig. 5:** Optical Microscope Image (600 X): (a)Sample Prepared at (450 $^{o}$C , 60 min. , 40 MPa and 20 wt. % NaCl), (b) Sample Prepared at (650 $^{o}$C , 180 min. , 80 MPa and 40 wt. % NaCl)

## 3.2. Input Manufacturing Parameters Effect on Responses

The experiments were carried out to find the effect of the pore-forming agent (NaCl wt. %), compacting pressure ,sintering temperature and sintering duration on the responses (porosity, coefficient of permeability and micro- hardness) for porous sintered tin-bronze alloy. Three levels for the fabricating parameters were considered Central Composite Design (CCD) during response surface methodology (RSM) was used .Table IIproves the control-parameters, designation and levels.

**Table 2:** Input Manufacturing Parameters and Their Levels

| Coded Factors | Actual Factors | Parameters | Unit | Actual levels | | |
|---|---|---|---|---|---|---|
| A | $P_{Ag}$ | Pore Agent | Wt. % | 20 | 30 | 40 |
| B | $p_c$ | Compact Pressure | MPa | 40 | 60 | 80 |
| C | T | Sintering Temperature | $^{o}$C | 450 | 550 | 650 |
| D | t | Sintering Time | min. | 60 | 120 | 180 |

Table III established the results of the responses based on experimentally design. Regressions models develop to predicting the responses by the squares method. The difference among measured results and predict results based on models showed in Fig. 6. These figures indicates that the develop models capable to representing the system under the given experimental domain. The coefficient of multiples determinations for the develop-models have the values$R^2$=98.80 % ; $R^2$=95.68 % and $R^2$=97.15 %respectability .

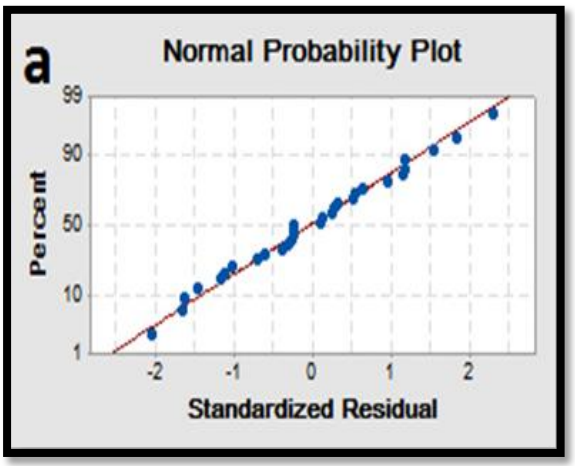
**P** = 1.58 + 2.125 A - 0.093 B + 0.0149 C - 0.0069 D + 0.00134 B*B+ 0.000213 D*D- 0.0055 A *B- 0.000661 A*C- 0.0011 A*D- 0.000313 B*D (2)

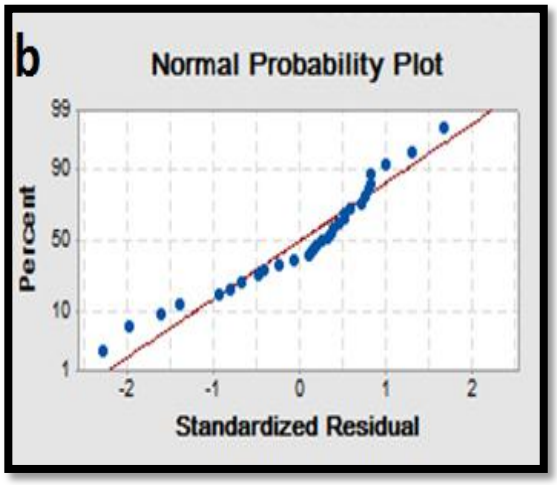
**K** = -0.003 + 0.00574 A - 0.00124 B+ 0.000033 C- 0.000011 D+ 0.000119 A*A+ 0.000024 B*B+ 0.000001 D*D- 0.000007 A*D-0.000002 B*C (3)

**Hv.** =73.4- 1.429 A + 0.1928 B+ 0.103 C- 0.1384 D- 0.000083 C*C+ 0.000476 D* D + 0.00085 A * D + 0.000064 B * D (4)

**Table 3:** Process Parameters and Experimental Data

| Pore agent (Nacl) (%) | Compact Pressure (MPa) | Temperature ($C^0$) | Time (min.) | Porosity % | Hardness Hv | K (Permeability coeffici.)* $10^{-3}$ cm/min. |
|---|---|---|---|---|---|---|
| 30 | 60 | 550 | 120 | 50.155 | 65.03 | 1.8078 |
| 40 | 80 | 650 | 60 | 63.026 | 53.01 | 2.6670 |
| 40 | 40 | 650 | 180 | 69.578 | 48.84 | 3.2148 |
| 20 | 80 | 450 | 60 | 37.125 | 77.93 | 0.8261 |
| 20 | 40 | 650 | 60 | 40.068 | 73.08 | 1.0776 |
| 30 | 60 | 550 | 120 | 52.236 | 60.50 | 1.8705 |
| 20 | 80 | 650 | 180 | 34.362 | 82.16 | 0.3143 |
| 40 | 40 | 450 | 180 | 71.183 | 45.32 | 3.4671 |
| 40 | 80 | 450 | 60 | 61.780 | 55.51 | 2.5593 |
| 30 | 60 | 550 | 120 | 53.083 | 60.86 | 1.8305 |
| 20 | 40 | 650 | 180 | 44.816 | 70.90 | 1.4817 |
| 30 | 60 | 550 | 120 | 51.688 | 64.01 | 1.8191 |
| 20 | 80 | 450 | 180 | 36.880 | 78.82 | 0.7184 |
| 40 | 80 | 650 | 180 | 59.270 | 56.46 | 2.3797 |
| 40 | 40 | 650 | 60 | 70.450 | 47.12 | 3.3675 |
| 40 | 40 | 450 | 60 | 74.336 | 41.67 | 3.6818 |
| 20 | 40 | 450 | 180 | 40.608 | 72.88 | 1.1225 |
| 20 | 80 | 650 | 60 | 35.865 | 81.13 | 0.5388 |
| 20 | 40 | 450 | 60 | 38.758 | 75.32 | 0.9878 |
| 40 | 80 | 450 | 180 | 36855 | 52.12 | 2.7114 |
| 30 | 60 | 550 | 120 | 51.750 | 61.51 | 1.3161 |
| 30 | 60 | 450 | 120 | 52.110 | 60.05 | 1.8858 |
| 40 | 60 | 550 | 120 | 66.134 | 50.34 | 2.9185 |
| 20 | 60 | 550 | 120 | 37.358 | 77.04 | 0.8531 |
| 30 | 60 | 550 | 120 | 48.719 | 68.85 | 1.1590 |
| 30 | 40 | 550 | 120 | 56.282 | 58.83 | 2.1552 |
| 30 | 80 | 550 | 120 | 46.091 | 69.01 | 1.5715 |
| 30 | 60 | 550 | 180 | 51.133 | 67.21 | 1.7511 |
| 30 | 60 | 550 | 60 | 51.701 | 65.88 | 1.8409 |
| 30 | 60 | 650 | 120 | 50.410 | 67.95 | 1.7241 |





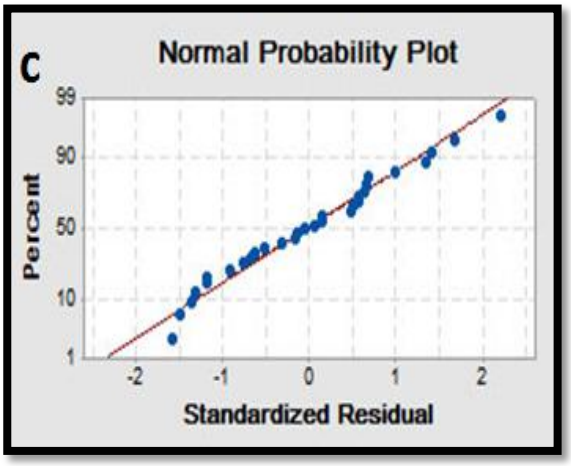




**Fig. 6:** Scatter Plot Diagram for: (a) P, (b) K(b) Hv

### 3.3. Parametric Analysis of Porosity

Porosity is one of the most important property to be studied because it's vital influence on the characteristics of porous structure.Fig. 7shows the main effect plot of each input parameter on the porosity P.

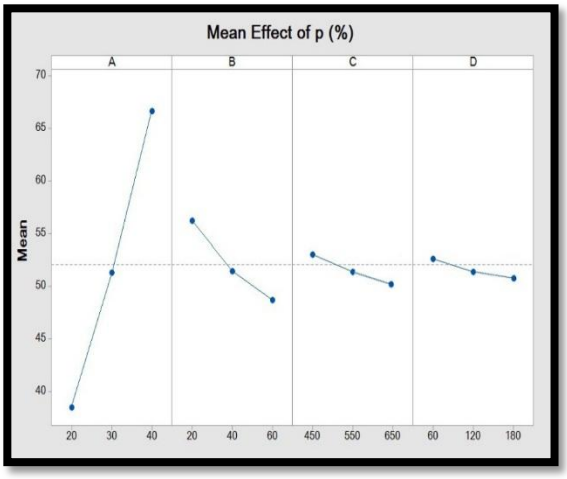



**Fig. 7:** Main Effect Plot for Means of The Porosity

P tends to increase with the increase in parameter A (NaCl wt. %) from (37.982 % to 67.215%). It can be attributed that the porosity is increased with increasing NaCl wt. % . The relationship is direct between them , since the particles of NaCldissolved at the last step of preparation process of the sample . At the dissolve of NaCl particles , the particle will leave a hole or pore. Some these pores are closed and isolated, but the most are interconnected and have an open ends. On the contrary, the main effect of parameter B (compacting pressure) displays a reverse tendency. The increase in this parameter causes decreases in the P about (56.863 % to 48.281%) with keeping the other parameters unchanged. Usually, the porosity decreases with the increase of compact pressure due to an increase in plastic deformation and reduction the voids between the particles , eventually increase in density [11].The analysis appears a small effect of sintering temperature and sintering time on porosity. The analysis shows a slight decreasing in the porosity from (53.957 % to 49.362 %) and (52.898 % to 50.624 %) with increasing sintering temperature and sintering time, respectively . this can attributed to the fact that increasing of these parameters lead to increase the diffusion between the particles and causes a reduction voids and pores and so increasing the density , hence decreases the porosity [12].

### 3.4. Parametric Analysis of Permeability Coefficient

Fig. 8 shows the main effect plots of the four studied parameters on permeability coefficient (K cm/min.).

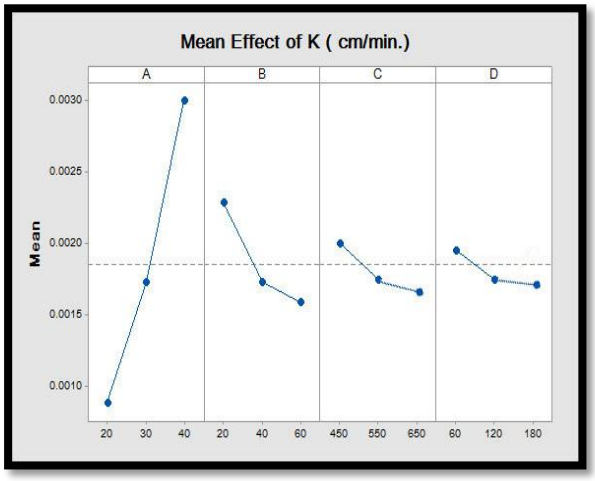



**Fig. 8:** Main Effect Plot for Means of K

It is obvious that (A and B) have significant impacts on permeability coefficient (K cm/min.). Permeability coefficient (K cm/min.) increases (from 0.0867 * $10^{-3}$ cm/min. to 0.3024 * $10^{-3}$ cm/min.), when A (NaCl wt. %) increases from 20 wt. % to 40 wt %.This is due to the fact that an increase in factor A causes the increase of porosity. So the higher the percentage of pores in the porous structure leads to increase the permeability through the porous media. The liquid (water in this study) is passed through pores connected through the porous bronze structure bythe results of Darcy's equation demonstrated in Table III .

Factor B (compact pressure) appears an opposite tendency. Permeability coefficient (K cm/min.) decreases from (0.2307 * $10^{-3}$ cm/min. to 0.1563 * $10^{-3}$ cm/min.) , when the compact pressure

increases from 40 MPa to 80 MPa . keeping the other parameters unchanged. It is clearly observed through Figure (4.14) and confirmed by the results shown in Table (4.2) that increasing the pressure negatively affects on the porosity .This leads to decrease the permeability coefficient.

The analysis shows that the permeability coefficient (K cm/min.) decreases with the sintering temperature and sintering time . It is clear from the main effect plot that each of these parameter variable displays a slight decreasing in K (cm/min.) about :(0.2027 * $10^{-3}$ cm/min. to 0.1683 * $10^{-3}$ cm/min.) and (0.1974 * $10^{-3}$ cm/ min. to 0.1748 * $10^{-3}$ cm/min.) with increasing sintering temperature (450 $C^{o}$ to 650 °C) and sintering time(60 min. to 180 min). This is because of an increase of these parameters lead to reduce size of the pores and contribute to the closure of the pores

in porous structure , this causes a decrease in the permeability throughout porous media [13].

### 3.5. Parametric Analysis of Micro-Hardness (Hv)

Fig. 9 illustrates the main effect plots of the controllable parameters on micro-hardness (Hv.) . The Micro-Hardness (Hv) decrease a from (76.926 to 50.172 Hv). with increasing factor A (NaCl wt. %) from 20 wt. % to 40 wt. % .

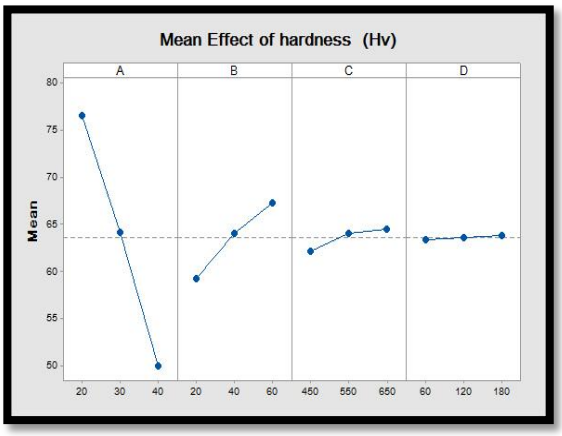
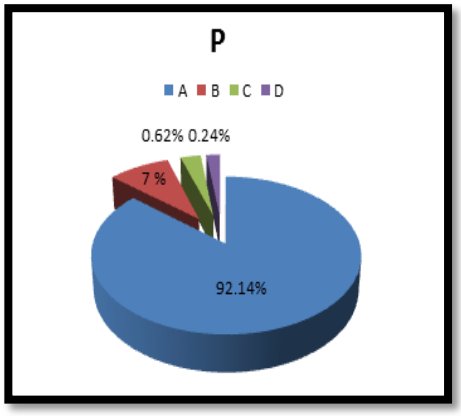
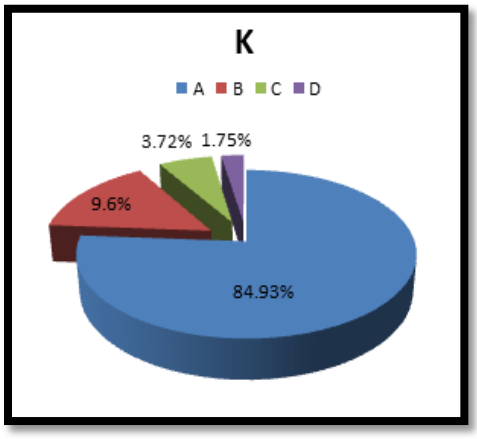



**Fig. 9:** Main Effect Plot for Means of Hv

Higher content of NaCl wt. % available at the porous tin-bronze alloy causes increasing in total percentage of pores on the structure thus, this causes a general weakness in the manufactured alloy because the high porosity acts as a riser of stresses in the structure and this leads to a decrease in micro-hardness of porous structure. Therefore, the optimal value of this parameter must be chosen to achieve the equilibrium between high permeability with good mechanical properties. It is clear from the main effect plot of factor B (compact pressure) that Hv increases from (58.893 to 67.792) with increases in compact pressure alones at constants mid.-values of other factors. This is because the increase in compact pressure causes an increase in the density resulting from plastic deformation and joining between the particles of powder which reduces the in the structure. Finally, selecting higher levels for factor C (sintering temperature) and factor D (sintering time) results in larger micro-hardness (Hv.) from (61.726 to 65.929) and (62.958 to 64.315) respectively. This is attributed to the diffusion processes which cause necks to form and grow at contact points of particles , reduce the porosity and promote the densification. So this causes increasing in mean density and micro-hardness (Hv.) for porous tin-bronze samples. That is supported by the results presented in Table III and Ref. [12].
From ANOVA analysis, the parameter of NaCl wt. % has a greater influence on the P (%), K (cm/min.) and Hv , followed by compacting pressure, sintering temperature and sintering time as shown Figures 10, 11 and 12.



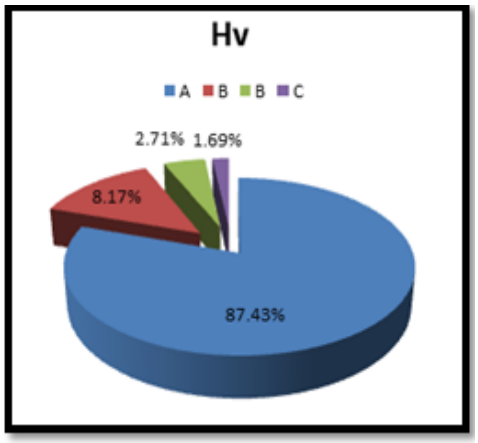




**Fig. 10:** Contribution Percentages of Process' Conditions on Responses: (a- Porosity, b- Permeability Coefficient and c-Micro-hardness)

### 3.6. Optimization of Manufacturing Conditions'

The responses optimizer option within the design of experiments modules of Minitab-statisticallsoftware'package, releases 17, was utilizing. It can be concluding that manufacturing processes, NaCl (pore agent) of (35.36 wt. %), (40 MPa) compact pressure , temperature of sintering (593.5 $^{o}$C) and (180 min.) time of sintering to gives maximum of porosity (P%), coefficient of permeability (K cm/min.) and micro- hardness (Hv). Responses within the satisfactory region.

### 3.7. Confirmations of the Optimums Results

The confirmations experiments is important steps and an indispensables parts of each optimizations attempts to validates the optimumsprocess'sconditions that result from responses surfaces methodology approaches'. Verifications experiments perform at the obtained optimally inputsparametric setting to comparing the actuals (P%), K (cm/min.) and (Hv) with the optimals responses get through desirability approach. Table IV summarizes the comparisons of the experimental the responses with their predicting values and their percentages of relatives verification errors by optimally manufacturing conditions'.

**Table 4:** Confirmations Tests Results and Percentages Error

| Response | P (%) | | K (cm/min.) | | Hv. | |
|---|---|---|---|---|---|---|
| | Exp. | Pred. | Exp. | Pred. | Exp. | Pred. |
| | 66.32 | 64.01 | $2.96*10^{-3}$ | $3.15*10^{-3}$ | 57.12 | 54.77 |
| Error % | | 3.48 | | 6.42 | | 4.11 |

The error percentage is with-in the ranges(3.48 to 6.42) %. So the developing predicting modeles establish through Minitab can be success full used to predicting pore agent (NaCl wt. %), compact

pressure, temperature and sintering time on for any collection of the porosity (P %), coefficient of permeability (k) and micro-hardness (Hv) values at the ranges of the conduct experiments.

## 4. Conclusion

When increase the percentage addition of NaCl wt. % , decrease compacting pressure, decrease sintering temperature and sintering time leads to an increases percentage of the porosity and permeability , but decrease in micro-hardness of porous tin-bronze alloy .

The optimal setting of process parameters that can be used in fabricating of (porous sintered 10 wt% tin-bronze alloy) according to response surface methodology analysis results are: pore- forming agent (35.36 wt. % NaCl), compact pressure (40 MPa), temperature of sintering (593.5 °C) and (180 min.) as time of sintering to get maximum of porosity (66.32 %) , permeability coefficient (2.96*10-3 cm/min.) and micro-hardness (57.12 Hv) .

From ANOVA analysis, the parameter of NaCl wt. % has a greater influence on the P (%), K (cm/min.) and Hv, followed by compacting pressure, sintering temperature and sintering time.

Responses surfaces methodology (RSM) can be used to developsmathematical models for predictions of mechanicals and physicalsproperties to porous structure of sintered tin-bronze alloys with-inthe parameters ranges of this study.

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