

**Synthesis of Antibacterial  
Additives used in Metal  
Working Oils from  
Local Materials**

By

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# **PROBLEM OF THE RESEARCH**

(1)

It is known that one of the serious environmental problems is the Accumulation of plastic bottles made of "Polyethylene Terephthalate" (PET) in large quantities and the inability of natural factors to analyze them or get rid of them, due to (PET) has high thermal stability.

## (2)

Another environmental and health problem is the bi-products of the bacterial growth in cutting fluids. The contaminated cutting fluid has irritating effect on the workers skin, in addition to the bad smells produced.

# AIM OF THE WORK

**The objectives of this study are:**

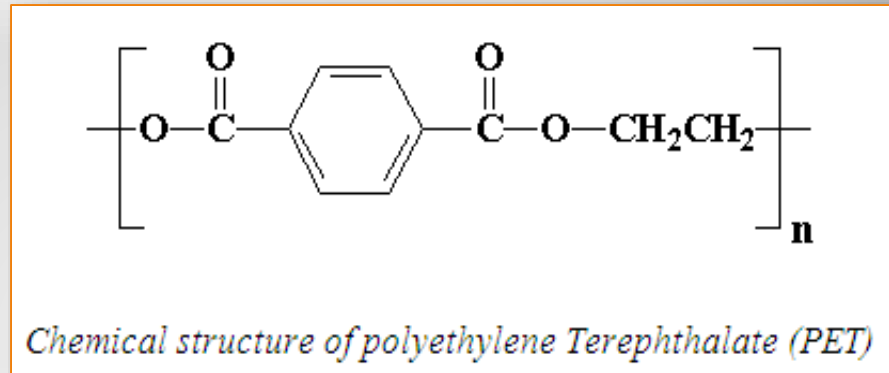
- 1-** Recycling the used polyethylene Terephthalate (PET) polymer.
- 2-** Preparation of new generation of biocides based on the recycled (PET).
- 3-** Using the prepared biocides to improve the stability of the cutting fluids against the bacterial growth.
- 4-** Retain the Tribological properties of the cutting fluids after the addition of the prepared biocides.
- 5-** Evaluation of the cutting fluids after the addition of the prepared biocides in industrial scale.



**EXPERIMENTAL**

# 1. Materials

## a. PET Polymer



- PET polymer was collected as waste products of water bottles and other fabricates signed by (PET) label. Then the products were washed, grinded and packed for use.

# 1. Materials

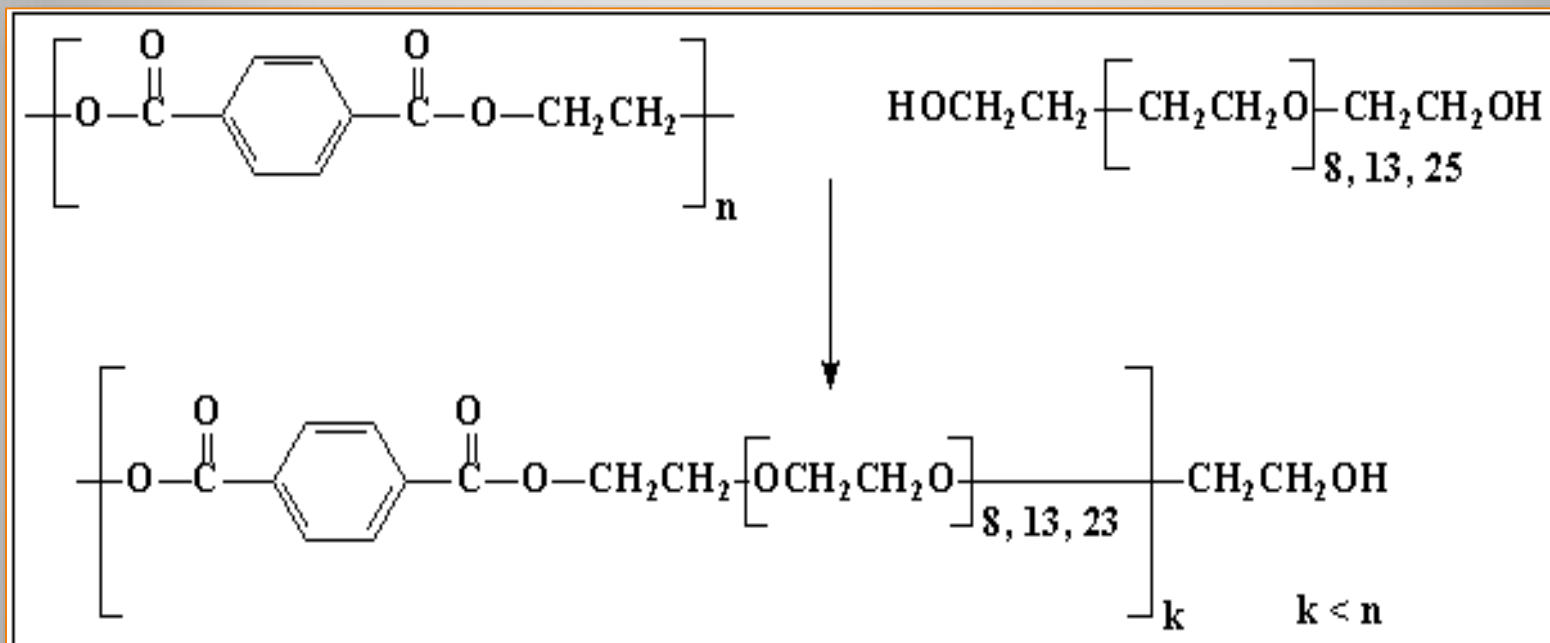
## b. Chemicals

- The chemicals used in the study were listed in the following table →

Compound	Purity	Supplier
Acetone	AR	ADWIC
Petroleum ether	AR	ADWIC
Xylene	AR	ADWIC
Bromoacetic acid	AR	BDH
Polyethylene glycol-400	AR	SIGMA
Polyethylene glycol-600	AR	SIGMA
Polyethylene glycol-1000	AR	SIGMA
p-toluene Sulfonic acid	AR	SIGMA
Pyridine	AR	SIGMA
Triethyl amine	AR	SIGMA

## 2. Synthesis of Biocides

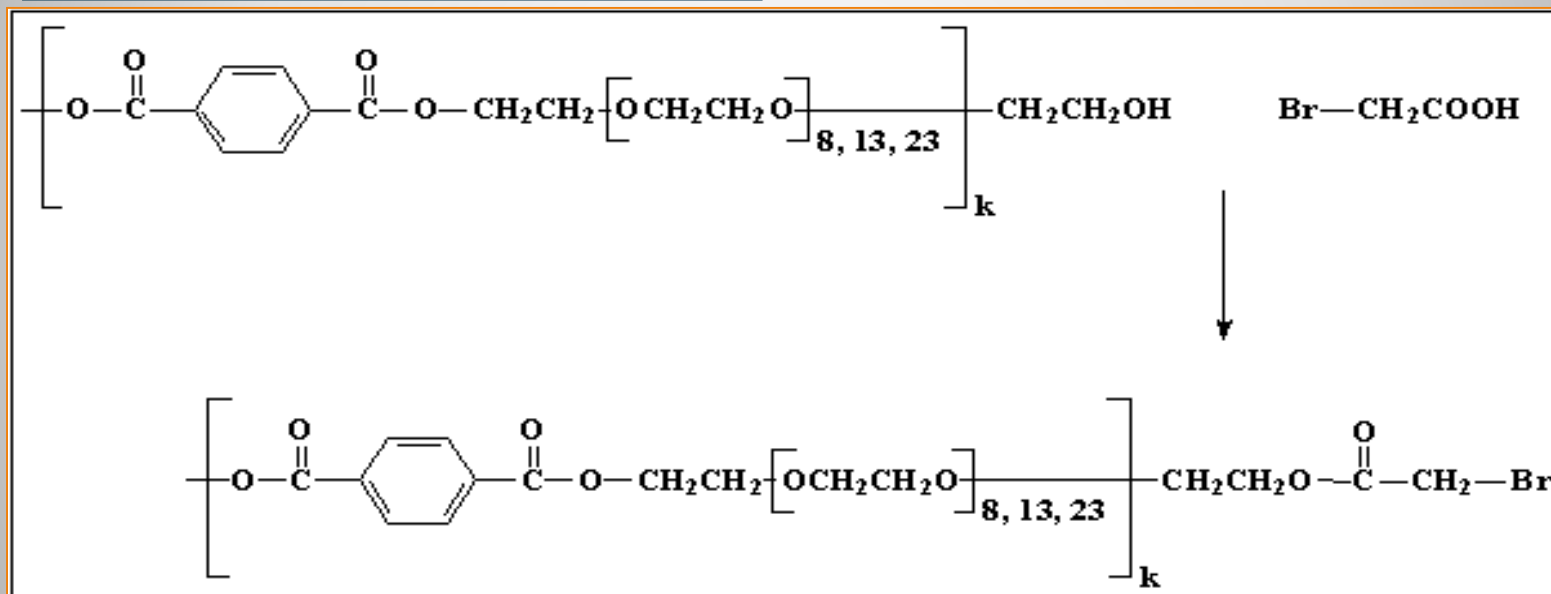
### a. Hydrolysis of PET Polymer



The obtained products were abbreviated as (PET400, PET600 and PET1000).

## 2. Synthesis of Biocides

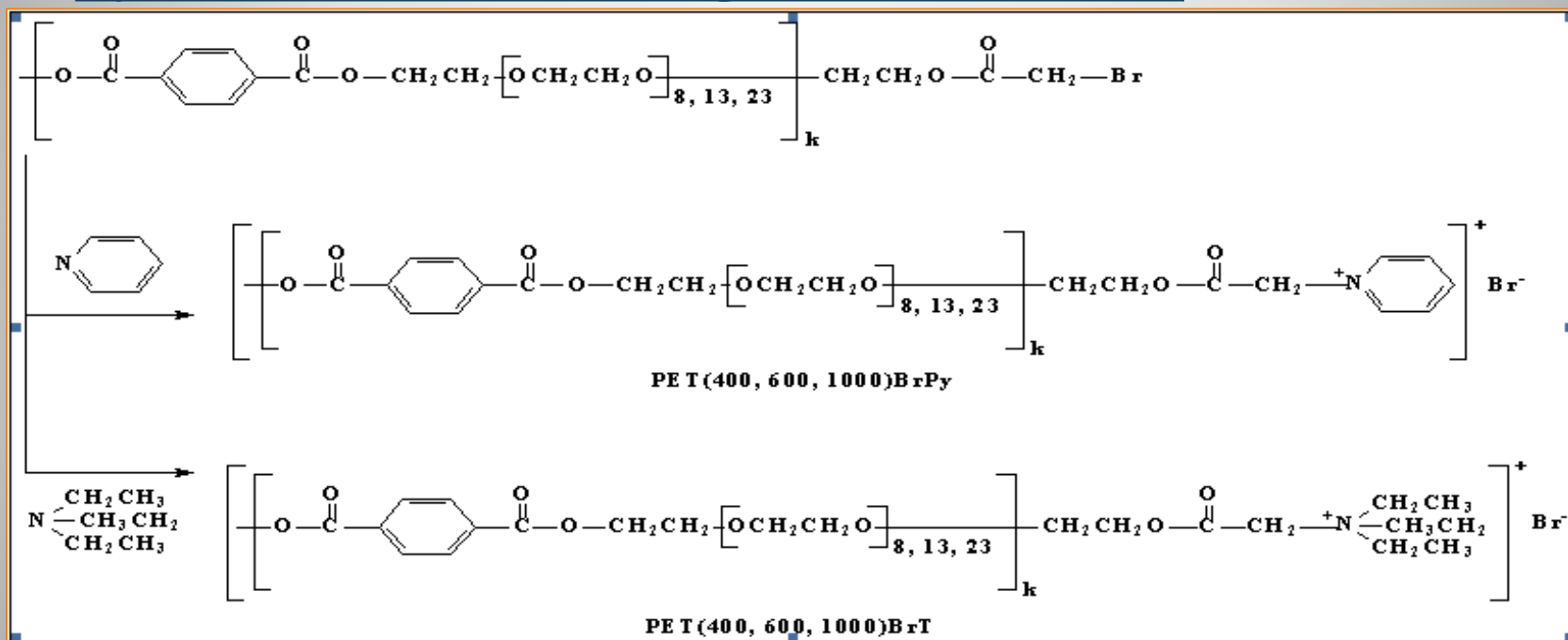
### b. Synthesis of Bromoacetate Derivatives (Esterification Process)



The obtained products were abbreviated as (PET400Br, PET600Br and PET1000Br).

## 2. Synthesis of Biocides

### c. Synthesis of the Cutting Fluid Additives



The obtained products were abbreviated as (PET400BrPy, PET600BrPy & PET1000BrPy for pyridine derivatives and PET400BrT, PET600BrT & PET1000BrT for Triethylamine derivatives).

### **3. Formulation**

- **The cutting fluids used in this study were two types; the first is the commercial cutting fluid and the second is the formulated cutting fluid .**

## 3. Formulation

### a. The Commercial cutting fluid

- It is formed of water (100 ml), base oil (3.75 ml), emulsifier (0.825 ml), corrosion inhibitor (0.045 ml), oilness agent (0.25 ml), coupling agent (0.075 ml) and biocide (Glokill 77 = 0.055 ml).



## 3. Formulation

### b. The Formulated cutting fluid

- It is formed of water (100 ml), base oil (3.75 ml), emulsifier (0.825 ml), corrosion inhibitor (0.045 ml), oiliness agent (0.25 ml), coupling agent (0.075 ml) and the synthesized additives (PET400BrT, PET600BrT, PET1000BrT, PET400BrPy, PET600BrPy & PET1000BrPy) which added individually with different ratios (0.05, 0.025, 0.0125, 0.00625 ml).

## 4. Evaluation of Synthesized Additives

The synthesized additives were evaluated as:

- First, The Biocidal activity assay of the additives against the microorganisms in the cutting fluids,
- Second, describing the tests of the different Tribological properties of the cutting fluids during metal working processes .

## 4. Evaluation of Synthesized Additives

### a. The Biocidal Activity Assay

- The Biocidal activity of the synthesized additives (PET400BrT, PET600BrT, PET1000BrT, PET400BrPy, PET600BrPy and PET1000BrPy) was evaluated using freshly prepared cutting fluid contaminated with bacteria in presence of the (0.1 - 1%) of the different additives.

## 4. Evaluation of Synthesized Additives

### b. The Tribological Properties

- The tribological properties of the cutting fluids formulated by using the synthesized additives (PET400BrT, PET600BrT, PET1000BrT, PET400BrPy, PET600BrPy and PET1000BrPy) were compared to the commercial cutting fluid formulated using the commercial biocides, corrosion inhibitor and emulsifier, by using (ASTM) and (IP) standards. The additives of the cutting fluids must not change the tribological properties of the cutting fluids.

## 4. Evaluation of Synthesized Additives

The Tribological properties of the cutting fluids include :

Tests	Standard Methods
Kinematic Viscosity at 40 °C	ASTM D 445
Kinematic Viscosity at 100 °C	ASTM D 445
Viscosity Index	ASTM D 2270
Specific Gravity @ 15 / 4 °c	ASTM D 4052
Rust Prevention	ASTM D 665
Open Flash Point (Pensky-Martens Method)	IP 35
Emulsification Power	IP 263
Surface Tension	Hafiz, Badawi, El-Deeb, Soliman & El-Awady, 2010
pH Value	Reported

# RESULTS & DISCUSSION

## 1. Confirmation of the Structures of the Synthesized Additives and Intermediates

- The chemical structures of the synthesized additives and the intermediates were confirmed using (FT-IR) and ( $^1\text{H}$ NMR) spectra.

## 2. Evaluation of the Synthesized Additives

### a. The Biocidal Activity Assay (Antimicrobial Evaluation)

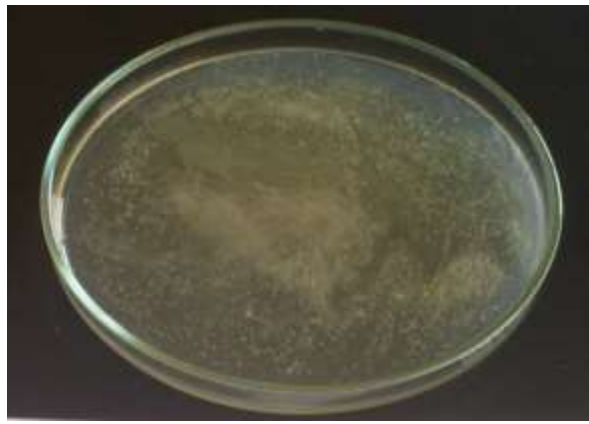
- The synthesized additives are considered as cationic quaternary compounds, due to the presence of quaternary ammonium nitrogen atoms in their chemical structures. The quaternary ammonium compounds are considered as efficient biocidal agents, which defeat the growth of different bacterial strains.



## 2. Evaluation of Synthesized Additives

The results of the biological activities of the different synthesized additives as biocides in the contaminated cutting fluids were listed in the following table:

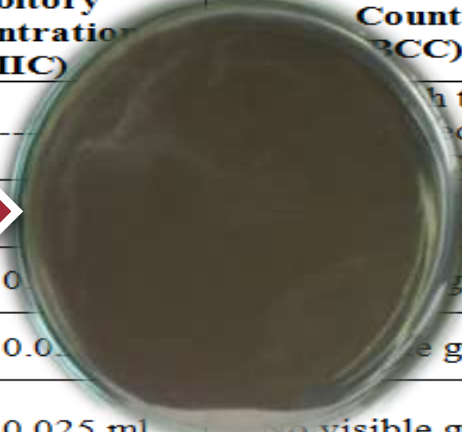
Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count (BCC)	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	Too much to be counted (TMTC)	-----
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with		= 0.025 ml	No visible growth	About 100%
		= 0.025 ml	No visible growth	About 100%
		= 0.025 ml	No visible growth	About 100%
		= 0.025 ml	No visible growth	About 100%
		= 0.025 ml	No visible growth	About 100%



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Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count (BCC)	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	h to be d )	-----
Cutting Fluid with (PET400BrT) Additive			growth	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0	growth	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.0	growth	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%



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The results of the biological activities of the different synthesized additives as biocides in the contaminated cutting fluids were listed in the following table:


Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	High bacterial growth	-----
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%



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The results of the biological activities of the different synthesized additives as biocides in the contaminated cutting fluids were listed in the following table:

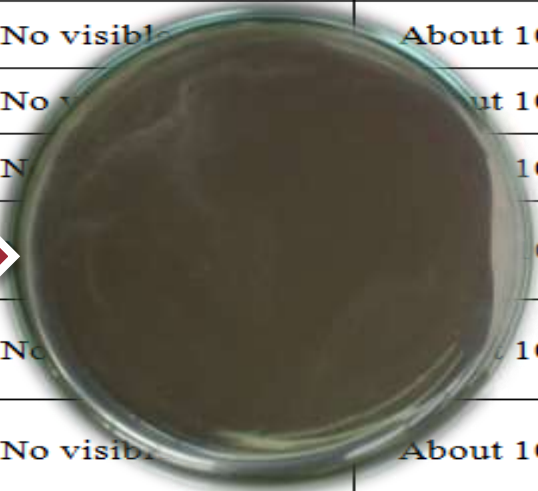
Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count (BCC)	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	Too many	-----
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%



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Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count (BCC)	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	Too much to be counted (TMTC)	-----
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0.025 ml	No visible	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.025 ml	No visible	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible	About 100%
Cutting Fluid with (PET600BrPyr) Additive		0.5% = 0.025 ml	No visible	About 100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible	About 100%

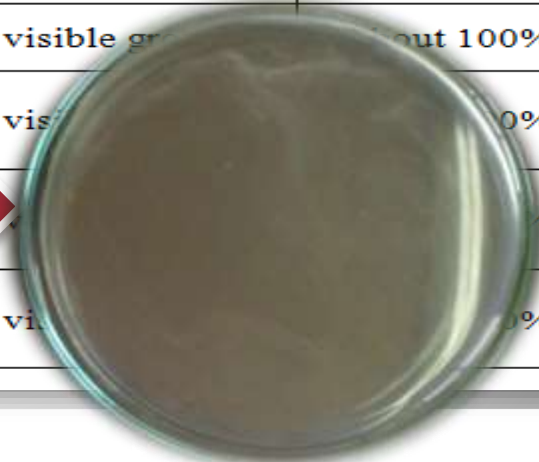




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Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Colonies Count (BCC)	Efficiency of additive (%)
Cutting Fluid without Antibacterial Additive		-----	Too much to be counted (TMTC)	-----
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible growth	100%
Cutting Fluid with (PET600BrPyr) Additive				100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible growth	100%



## 2. Evaluation of Synthesized Additives

The results of the biological activities of the different synthesized additives as biocides in the contaminated cutting fluids were listed in the following table:

<b>Samples</b> \ <b>Tests</b>	<b>Minimum Inhibitory Concentration (MIC)</b>	<b>Bacterial Colonies Count (BCC)</b>	<b>Efficiency of additive (%)</b>
<b>Cutting Fluid without Antibacterial Additive</b>	-----	Too much to be counted (TMTC)	-----
<b>Cutting Fluid with (PET400BrT) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%
<b>Cutting Fluid with (PET600BrT) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%
<b>Cutting Fluid with (PET1000BrT) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%
<b>Cutting Fluid with (PET400BrPyr) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%
<b>Cutting Fluid with (PET600BrPyr) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%
<b>Cutting Fluid with (PET1000BrPyr) Additive</b>	0.5% = 0.025 ml	No visible growth	About 100%



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The results of the biological activities of the different synthesized additives as biocides in the contaminated cutting fluids were listed in the following table:

Samples	Tests	Minimum Inhibitory Concentration (MIC)	Bacterial Concentration (BCC)	Efficiency of additive (%)
Cutting Fluid with Antibacterial Additive		---	Too much to be counted (MIC)	---
Cutting Fluid with (PET400BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrT) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET400BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET600BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%
Cutting Fluid with (PET1000BrPyr) Additive		0.5% = 0.025 ml	No visible growth	About 100%

**THE OBTAINED MINIMUM INHIBITION CONCENTRATION (MIC) VALUES WERE 0.5% For All Of The Different Additives**



**From the obtained data of the Biocidal tests, it can be concluded that the synthesized additives are efficient in protecting the cutting fluids from the attack of the bacteria.**

**But to determine the exact concentration which will be suitable as an efficient additive, the results of the Tribological properties were considered.**

## b. The Tribological Properties

C.F. Type Tribol. Tests	The Commercial Cutting Fluid	The Formulated Cutting Fluid				Notes
		With 0.1% Synthesized Additives	With 0.25% Synthesized Additives	With 0.5% Synthesized Additives	With 1% Synthesized Additives	
Kinematic Viscosity at 40 °C	32.87	32.74 – 32.90	32.75 – 32.92	32.81 – 32.95	32.85 – 32.98	(cSt)
Kinematic Viscosity at 100 °C	5.39	5.37 – 5.39	5.37 – 5.41	5.38 – 5.41	5.39 – 5.42	(cSt)
Viscosity Index	96	96	96	96	96	-----
Specific Gravity @ 15/4 °C	0.9111	0.9110 – 0.9111	0.9110 – 0.9111	0.9110 – 0.9111	0.9111 – 0.9112	-----
Rust Prevention	- ve	- ve	- ve	- ve	- ve	-----
Open Flash Point (Pensky- Martens Method)	195	193 - 196	193 - 195	194 - 196	193 - 196	(°C)
Emulsificat ion Power	Emuls. stability is high (to 5 days)	Emuls. stability is very high (to 14 days)	Emuls. stability is very high (to 14 days)	Emuls. stability is high (to 7 days)	Emuls. stability is high (to 5 days)	Increasing the concentration of the additives to more than 1% decreases the emulsion stability of the cutting fluid formulation to less than 2 days
Surface Tension	-----	The synthesized additives are characterized by a high tendency towards lowering the surface tension of the water to lower values				-----
pH Value	9	8.97 – 9	8.95 – 9	8.95 – 9	8.90 – 9	-----

**From the obtained data of the Tribological properties, it can be concluded that increasing the ratio of the synthesized additives has no effect on several properties of the cutting fluids. The maximum additives concentration which has no effect on the stability of the cutting fluid formulation is 1%.**

# **Economical Study**

**- One Ton of the Imported Additive = 26315 LE.**

**- In case of the Synthesized Additives (PET400BrPy, PET600BrPy & PET1000BrPy) :**

<b>Materials</b>	<b>Cost</b>
<b>500 kg of PET</b>	<b>-----</b>
<b>500 kg of PEG</b>	<b>12500 LE</b>
<b>40 kg of Bromoacetic acid</b>	<b>2000 LE</b>
<b>0.7 kg of Pyridine</b>	<b>490 LE</b>
<b>Total = 1 Ton</b>	<b>Total = 14990 LE</b>
<b>+ 10 % Operating Expenses</b>	<b>16489 LE</b>
<b>+ 10 % Electricity &amp; Equipments</b>	<b>Final Cost = 17988 LE</b>

**- In case of the Synthesized Additives (PET400BrT, PET600BrT & PET1000BrT) :**

<b>Materials</b>	<b>Cost</b>
<b>500 kg of PET</b>	<b>-----</b>
<b>500 kg of PEG</b>	<b>12500 LE</b>
<b>40 kg of Bromoacetic acid</b>	<b>2000 LE</b>
<b>0.7 kg of Triethylamine</b>	<b>22.5 LE</b>
<b>Total = 1 Ton</b>	<b>Total = 14522.50 LE</b>
<b>+ 10 % Operating Expenses</b>	<b>15974.75 LE</b>
<b>+ 10 % Electricity &amp; Equipments</b>	<b>Final Cost = 17427 LE</b>

# Conclusion



**1-** The Recycling of the (polyethylene terephthalate) polymer can produce efficient additives to the cutting fluid formulation without changing the Tribological properties of these formulations according to the (ASTM) and (IP) Standards.

**2-** The synthesized additives have an excellent biocidal activity against the bacterial growth in the cutting fluids, so that these compounds can be used as efficient biocides for preventing the bacterial growth in the cutting fluids.

**3-** Increasing the ratio of the synthesized additives has no effect on several properties of the cutting fluids including: kinematic viscosity, viscosity index, specific gravity, flash point, rust prevention and pH value.

**4-** The maximum additives concentration which has no effect on the stability of the cutting fluid formulation is 1%.

**5-** The synthesized additives have lower cost than the imported additive which leads to increase the percentage of Local Manufacturing.

T H A N K Y O U

*Thank you very much!!*

*Thank You*

