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Hansen Solubility parameters and Green Solvents for Organic Photovoltaics

[a]Defaru Negera and [b]Teketel Yohannes (Prof.)
[a]Department of Applied Chemistry, School of Applied Science, Adama Science & Technology University, Ethiopia
[b]Department of Industrial Chemistry, College of Natural Science, Addis Ababa Science and Technology University, Ethiopia

ABSTRACT

The determination and prediction of solubility behavior of organic semiconductors to use them is very important.[1] The concept Hansen solubility parameters is applied for the study. HSPs for $PC_{61}BM$ were determined using HSPiP software. In this experiment, we used 20 and 39 solvents in the first and second phases of the experiment respectively to determine HSPs for $PC_{61}BM$. The results obtained were 18.23, 3.75, and 4.51$MPa^{1/2}$ for dispersive, polar and hydrogen bonding for the first and 17.58, 3.73 and 4.79 $MPa^{1/2}$ for the second respectively. These results were compared to HSPs of chloroform, Limonene and Benzaldehyde. Limonene is used for cleaning in the electronic and printing industries, and in paint as a solvent. [2] It was selected as a solvent to replace the chlorinated type solvents. (HSPs) of Limonene, with δD, δP and δH of 17.20, 1.8 and 4.3 $MPa^{1/2}$ respectively, were obtained from the HSPiP list of solvents and the calculated Relative Energy Difference of 0.333 for Limonene to $PC_{61}BM$ suggested that limonene could be a good non-chlorinated for solution processing of fullerene-based polymer solar cells. The Limonene processed active layer in this work displayed a maximum power conversion efficiency of 3.19 % and our results suggest that Limonene would be a promising solvent for environment – friendly fabrication of polymer solar cells if more efforts is done to improve the power conversion efficiency.

Key Words: $PC_{61}BM$, Dispersive, Polar Bond, Hydrogen Bond, Limonene, Hspip, Photovoltaics.

1. INTRODUCTION

The growing need for energy and inadequate fossil fuel resources on this world need the growth of more sources of renewable energy. [3] To reduce this difficulty, people have been working on solar energy conversion to electricity for years [3] But this cannot be achieved only through Si-based solar cell manufacturing, which is expensive and the main source of solar electricity. [4] This requires alternative sources of solar electricity to be developed and produced [4]. In this case the finding of conducting polymers, the production of OPV devices (organic solar cells) becomes promising.

The main advantages of OPV devices compared to classic solid-state photovoltaic device are; the cost of fabricating OPV device are cheaper, the devices are lightweight, the devices are flexible, and the shape can be customized, and the device can be semitransparent. [5]

The charge transfer from conjugated polymers to fullerene molecules when photon is allowed through the bulk heterojunction of the components is the base for the most organic solar cells of today. [6-10] The Single-Junction organic solar cells with efficiency of (7 – 8) % was reported as the best. [11] The silicon devices gave 15 and 20 % efficiency for amorphous and crystalline respectively.[12,13] But, the organic solar cells still are well known with their low efficiency. [14] This is because the organic components, the blend of donor - acceptor and solvent interaction effect during the solution processing, on film morphology formation is not clearly understood.[15] Many studies show that short circuit density (Jsc) and open circuit voltage (Voc) are mainly affected by the nanostructure of the film morphology.[16-18]

In the same way, the fabrication of polymer solar cells is also affected by the solubility of the components of the blend. [19] Solubility is the key factor in the growth of organic devices. The basis for this work is the concept introduced by Hildebrand and Scott in 1950. [20] Solvents and organic molecules were assigned a value allowing prediction of their solubility. According to this prediction, solvents and organic compounds used in the solar cells of similar cohesive energy density (E/v) would be more miscible. [21] This Hildebrand solubility parameter, δ is defined by eq 1

$$\delta \boxed{?} (E/V)1/2 \qquad 1$$

But this definition is not complete due to more than one ways of interactions between donor, acceptor and solvent materials. These are dispersiveforces, polar interactions and hydrogen bonding interactions. In 1967, Charles Hansen proposed that each type of interaction can be given an individual parameter. [22] He formulated three solubility parameters for evaluating polymers.[23] These interactions thus correlate to the dispersive solubility parameter (δD), the polar solubility parameter (δP), and the H-bonding solubility parameter (δH), respectively. The relationship between the HSPs and the cohesive energy density is shown by eq 2 below.

$$\delta^2 = (E/V) = \delta^2_D + \delta^2_P + \delta^2_H \qquad 2$$

The equation shows that each organic molecule can be represented by a coordinate in 3-dimensional space. It is called Hansen space, in which the three Cartesian units are replaced with three Hansen solubility parameters. When the distance between the two molecules is close in Hansen space, the miscibility of the molecules is expected to be high. This distance is known as the radius of interaction, R_A. The extent of miscibility of the materials can be explained in terms of relative energy difference (RED) that can be defined by the following equation, [24]

$$\mathrm{RED} = R_A/Ro \qquad 3$$

where Ro is the radius of the Hansen space or the radius of the sphere. Good solvents are expected to be found within this radius. The smaller the value of relative energy difference that approaches zero indicating the materials good miscibility whereas when RED is equal to 1 intermediate miscibility and when greater than 1, taken as no miscibility or the materials do not mix well. [21]

This technique has been used in many industries and research areas, to estimate the solubility and miscibility of organic materials, and resulted in obtaining Hansen solubility parameters values for many solvents and polymers. [19,22,24] We used this principle for [6,6]-phenyl-C_{61}-butyric acid methyl ester (PC60BM, Poly [2,3-bis-(3-octyloxypenyl) quinoxaline-5,8-diyl-alt-thiophene-2,5-diyl] (TQ1) and [6,6]-phenyl-C_{71}-butyric acid methyl ester (PC70BM) Hansen solubility parameters determinations.

The other issues of this work are about suitable solvents for the polymer solar cell application which currently are very limited, and till now the efficient polymer solar cells have been using chlorobenzene. [25] and dichlorobenzene [26,27] as solvent for the solution processing. But these solvents are toxic and hazardous to human health and environment. Thus, we need solvents of low toxicity or no toxicity for polymer solar cell solution processing, mainly for commercialization.

Many works have been attempted to bring new solvent systems for polymer solar cells solution processing of active layers. [28] The solvents, toluene, [29] xylene, [30] trimethylbenzene(TMB), [31] n-butylbenzene, and some others and related mixed once, [32] have been used for many BHJ materials but aromatic solvents are still hazardous. Therefore, green solvents are highly desirable for the solution processing of polymer solar cells, mainly for commercialization. In this case, for this work we used Limonene and benzaldehyde as solvents for processing the solar cell device fabrication and obtained somehow promising results that invites researchers for more effort in the area.

Limonene is a colourless liquid at room temperature. The structural formula for limonene is given below. d-limonene is naturally occurring in fruit. It is also common in cosmetic products, as a solvent for cleaning purposes, and as a flavouring agent. The empirical formula is $C_{10}H_{16}$, the molecular weight (136.23), boiling point (175.5 – 176 C), density (g/cm3 at 20 C^0) is 8.411 and vapour pressure (pa at 20 C^0) is 190.

The other solvent used was Benzaldehyde. Benzaldehyde is generally regarded as a safe food additive in the United States and is accepted as a flavouring substance in the European Union. The empirical formula is C_7H_6O, the molecular weight (106.12), boiling point (179.2 C^0), density (g/cm3 at 20 C^0) is 1.0415 and vapour pressure (pa at 20 C^0) is 130.

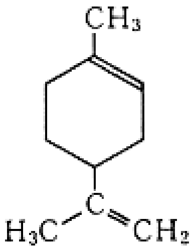

Fig 1. Chemical structure of Limonene

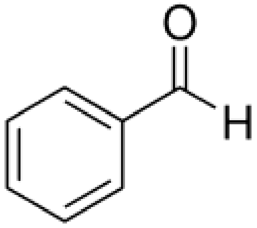

Fig 2 Structure of Benzaldehyde

2. EXPERIMENTAL DETAILS

2.1 Materials and instrumentations

The polymer solar cell devices were fabricated with a configuration of ITO/PEDOT: PSS/TQ1:PC61BM (1:3, w/w) /LiF/Al. Patterned indium tin oxide (ITO)-coated glass with a sheet resistance of 10 Ohm/square was used. It was cleaned under a wet-cleaning process inside ultrasonic bath, which was cleaned in isopropanol in an ultrasonic bath for 60 min and subsequently UV-ozone treated for 20 minutes. The solution of Poly [2,3-bis-(3-octyloxypenyl) quinoxaline-5,8-diyl-alt-thiophene-2,5-diyl] (TQ1) was prepared in Limonene and Benzaldehyde with a concentration of 20 mg/ml in a brown vial of 4 ml volume in a glove box. In the same way, the [6,6]-phenyl-C_{60}-butyric acid methyl ester (PC60BM) solution was prepared in a glove box with the concentration of 20 mg/ml in the solvents in a condition to achieve the TQ1:PC60M (1:3) ratio. Then, blend solutions were made by mixing the indicated ratio of the solutions and kept on the plate of temperature 50 – 60 degrees being stirred for 12 hours in the glove box. Blends of TQ1-PC60BM in 1:3 weight/weight ratio were prepared in Limonene and Benzaldehyde. The active layer was deposited by spin coating in a protected N_2 atmosphere (<0.1 ppm O_2, <0.1 ppm H_2O inside a glove box (M. Braun Intergas-systeme GmbH).

After spin coating the active layer, the samples were transferred into the vacuum chamber of the thermal evaporator (Univex 350 G, Oerlikon Lybold Vacuum GmbH) integrated within the glove box. LiF was deposited at the rate of 0.3 Ås^{-1} to the thickness of 0.3 nm first and then 100 nm Al was deposited with a deposition rate of 1 Å s^{-1} (Al) at a 10^{-6} mbar.

3. RESULTS AND DISCUSSION

Hansen Solubility parameters for [6,6]-phenyl-C_{61}- butyric acid methyl ester ($PC_{60}BM$) were determined. In the fabrication of conjugated polymer solar cells involving solution processing, the solvent is the key. Hansen solubility parameter determination is the principle that enables to determine dispersive, polar and hydrogen bonding interaction forces for polymers and fullerenes. The solubility tests for $PC_{61}BM$ were performed in the laboratory. The absolute solubility of $PC_{61}BM$ was recorded in ~ 20, 39 solvents with different HSP values. We dissolved 1mg of $PC_{61}BM$ in 1 ml of solvent, for each solvent. 1 for yes, 0 for no were recorded regarding whether each solvent dissolved the $PC_{61}BM$ at the chosen concentration of 1 mg/ml. This information was entered HSPiP and generated HSP and solubility spheres for $PC_{61}BM$. In this experiment, we used 20 solvents to determine the parameters for $PC_{61}BM$ in the first phase of the experiment. The results obtained were 18.23, 3.75, 4.51 for dispersive, polar and hydrogen bonding respectively. In the same manner 1mg/ml of 39 solvents were taken and 17.58, 3.73 and 4.79 were obtained. These results were compared to results of the Hansen solubility parameters of some solvents like chloroform, Limonene and Benzaldehyde.

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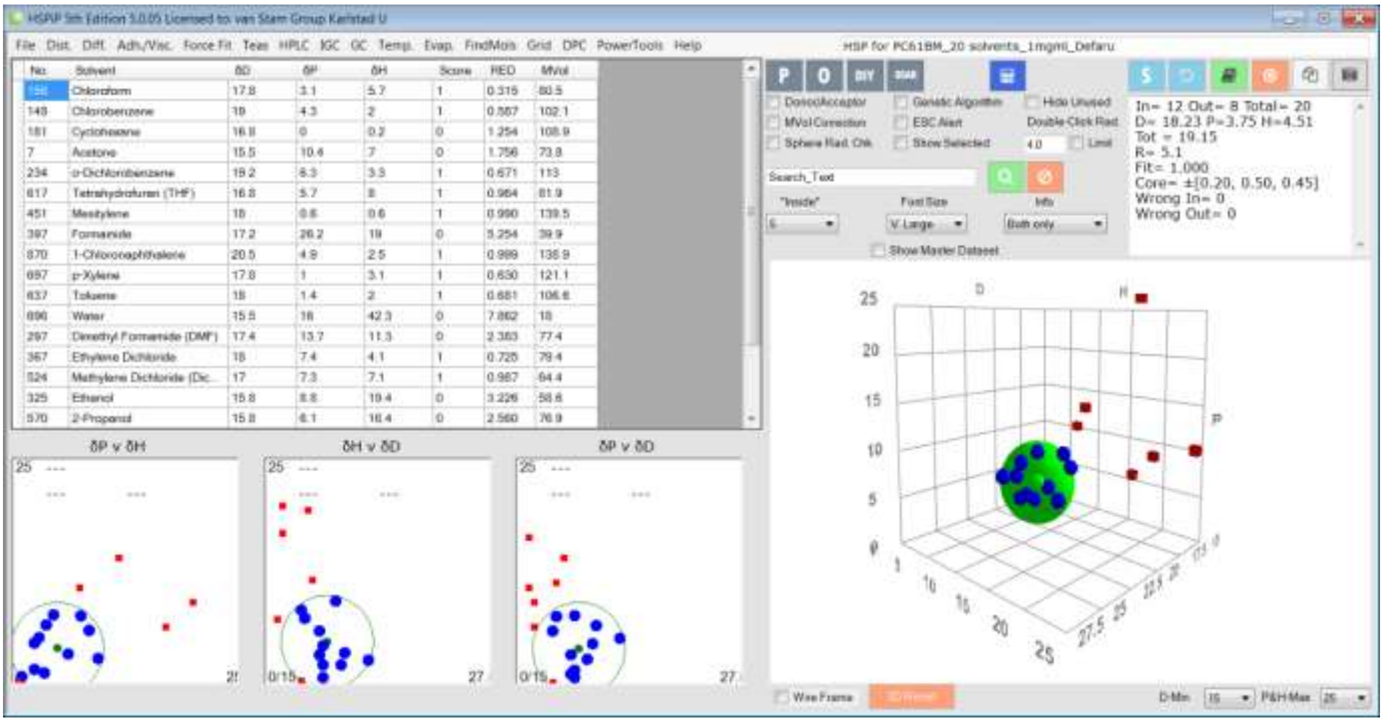
$PC_{61}BM$ Solubility test in different solvents

3.1 HSP and Solubility spheres for PC61BM _20 solvents_ 1 mg/ml

Table 1 Hansen solubility parameters for PC61BM compared to the parameters of some solvents

Material	D	P	H	Remark
$PC_{61}BM$	18.23	3.75	4.51	
TQ1	18.19	2.12	6.17	
Chloroform	17.8	3.1	5.7	
Limonene	17.20	1.8	4.3	
Benzaldehyde	19.4	7.4	5.1	



No.	Solvent	δD	δP	δH	Score	RED	MVol
156	Chloroform	17.8	3.1	5.7	1	0.316	80.5
148	Chlorobenzene	19	4.3	2	1	0.587	102.1
181	Cyclohexane	16.8	0	0.2	0	1.254	108.9
7	Acetone	15.5	10.4	7	0	1.756	73.8
234	o-Dichlorobenzene	19.2	6.3	3.3	1	0.671	113
617	Tetrahydrofuran (THF)	16.8	5.7	8	1	0.984	81.9
451	Mesitylene	18	0.6	0.6	1	0.990	139.5
397	Formamide	17.2	26.2	19	0	5.254	39.9
870	1-Chloronaphthalene	20.5	4.9	2.5	1	0.989	136.9
697	p-Xylene	17.8	1	3.1	1	0.630	121.1
637	Toluene	18	1.4	2	1	0.681	106.6
696	Water	15.5	16	42.3	0	7.802	18
297	Dimethyl Formamide (DMF)	17.4	13.7	11.3	0	2.383	77.4
367	Ethylene Dichloride	18	7.4	4.1	1	0.725	79.4
524	Methylene Dichloride (Dic...	17	7.3	7.1	1	0.987	64.4
325	Ethanol	15.8	8.8	19.4	0	3.226	58.6
570	2-Propanol	15.8	6.1	16.4	0	2.560	76.9

```
In= 12 Out= 8 Total= 20
D= 18.23 P=3.75 H=4.51
Tot = 19.15
R= 5.1
Fit= 1.000
Core= ±[0.20, 0.50, 0.45]
Wrong In= 0
Wrong Out= 0
```

Fig 3 Hansen solubility sphere of PC61BM generated using HSPiP software in 20 solvents

As it can be seen from the generated Hansen solubility sphere of PC61BM using HSPiP software eight solvents are out of the sphere usually are said non-solvents (red colored squares) and 12 solvents in inside the sphere are called good solvents (blue colored squares).

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Table 2 shows the list of 20 solvents with their corresponding Hansen solubility parameters, Score, RED and Mvol

No	Solvent	δD	δP	δH	Score	RED	Mvol
156	**Chloroform**	17.80	3.10	5.701	1	0.315	80.5
148	**Chlorobenzene**	19.00	4.30	2.00	1	0.587	102.1
181	**Cyclohexane**	16.80	0.00	0.2	0	1.254	108.9
7	**Acetone**	15.50	10.40	7.00	0	1.756	73.8
234	**O-Dichlorobenzene**	19.20	6.30	3.30	1	0.671	113
617	**Tetrahydrofuran (THF)**	16.80	5.70	8.00	1	0.894	81.9
451	**Mesitylene**	18.00	0.60	0.60	1	0.990	139.5
397	**Formamide**	17.20	26.20	19.00	0	5.254	39.9
870	**1-Chloronaphthalene**	20.50	4.90	2.50	1	0.999	138.9
697	**P-xylene**	17.80	1.00	3.10	1	0.630	121.1
637	**Toluene**	18.00	1.40	2.00	1	0.681	106.6
696	**Water**	15.50	16.00	42.3	0	7.862	18
297	**Dimethyl Formamide (DMF)**	17.40	13.70	11.30	0	2.383	77.4
367	**Ethylene Dichloride**	18.00	7.40	4.10	0	0.725	79.4
524	**Methylene Dichloride**	17.00	7.30	7.10	1	0.987	64.4
325	**Ethanol**	15.80	8.80	19.40	0	3.226	58.6
570	**2-Propanol**	15.80	6.10	16.40	0	2.560	76.9
368	**Ethylene Glycol**	17.00	11.00	26.00	0	4.472	55.9
698	**O-Xylene**	17.80	1.00	3.10	1	0.630	121.1
1078	**Indene**	18.70	2.60	9.00	1	0.927	116.9

In= 12 Out= 8 Total= 20
D= 18,23 P=3,75 H=4,51
Tot = 19,15
R= 5,1
Fit= 1,000
Core= ± [0,20, 0,50, 0,45]
Wrong In= 0
Wrong Out= 0

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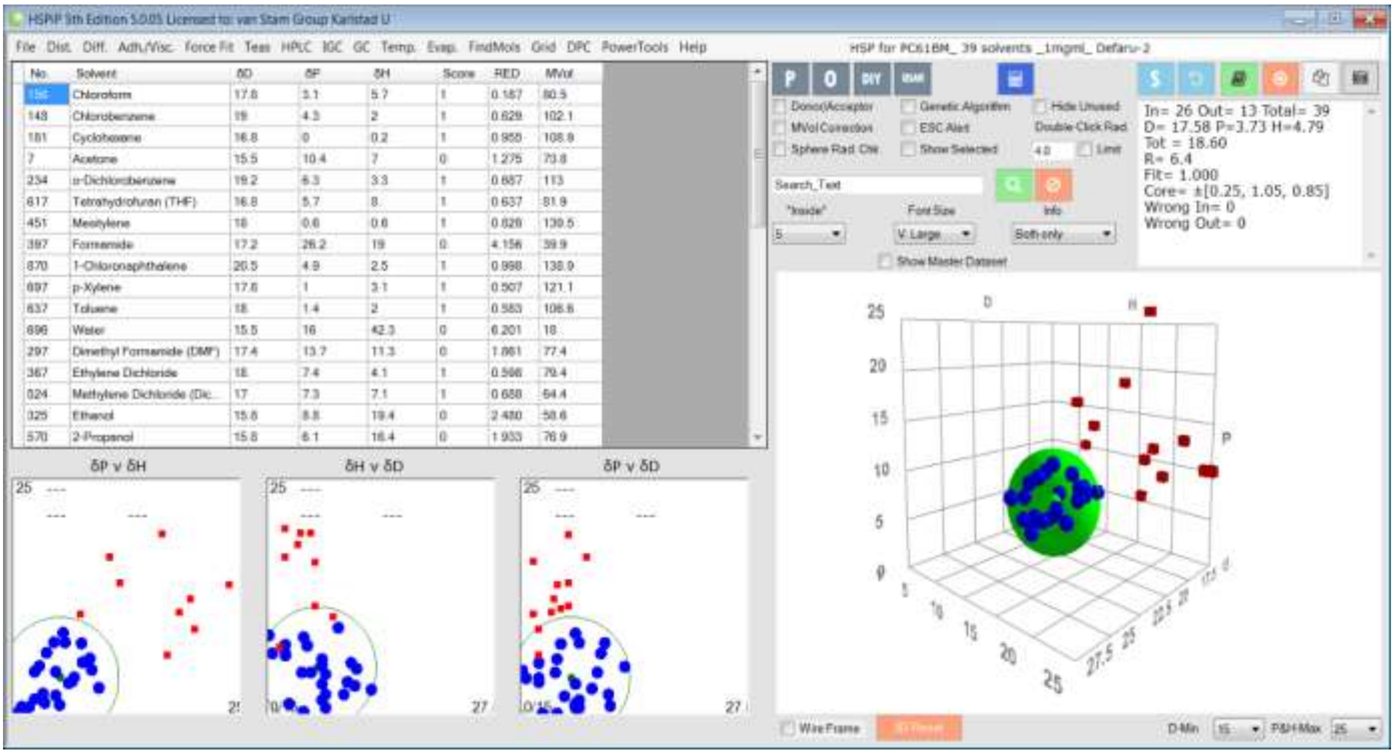
3.2 HSP and solubility spheres for PC61BM _39 solvents_ 1 mgml



No.	Solvent	δD	δP	δH	Score	RED	MVol
156	Chloroform	17.8	3.1	5.7	1	0.187	80.5
148	Chlorobenzene	19	4.3	2	1	0.629	102.1
181	Cyclohexane	16.8	0	0.2	1	0.958	108.9
7	Acetone	15.5	10.4	7	0	1.275	73.8
234	o-Dichlorobenzene	19.2	6.3	3.3	1	0.687	113
617	Tetrahydrofuran (THF)	16.8	5.7	8	1	0.637	81.9
451	Mesitylene	18	0.6	0.6	1	0.828	139.5
397	Formamide	17.2	26.2	19	0	4.156	39.9
870	1-Chloronaphthalene	20.5	4.9	2.5	1	0.998	138.9
697	p-Xylene	17.8	1	3.1	1	0.507	121.1
637	Toluene	18	1.4	2	1	0.583	106.8
696	Water	15.5	16	42.3	0	6.201	18
297	Dimethyl Formamide (DMF)	17.4	13.7	11.3	0	1.861	77.4
367	Ethylene Dichloride	18	7.4	4.1	1	0.598	79.4
524	Methylene Dichloride (Dic...	17	7.3	7.1	1	0.688	64.4
325	Ethanol	15.8	8.8	19.4	0	2.480	58.6
570	2-Propanol	15.8	6.1	16.4	0	1.933	76.9

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Info: Both only

In= 26 Out= 13 Total= 39
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Tot = 18.60
R= 6.4
Fit= 1.000
Core= ±[0.25, 1.05, 0.85]
Wrong In= 0
Wrong Out= 0

D-Min: 15
P&H-Max: 25

Fig 4 Hansen solubility sphere of PC61BM generated using HSPiP software in 39 solvents

As it can be seen from the generated Hansen solubility sphere of PC61BM using HSPiP software 13 solvents are out of the sphere usually are said non-solvents (red colored squares) and 26 solvents in inside the sphere are called good solvents (blue colored squares).

Tabel 3 shows the list of 39 solvents with their corresponding Hansen solubility parameters, Score, RED and Mvol

No	Solvent	δD	δP	δH	Score	RED	Mvol
156	Chloroform	17.80	3.10	5.701	1	0.315	80.5
148	Chlorobenzene	19.00	4.30	2.00	1	0.587	102.1
181	Cyclohexane	16.80	0.00	0.2	0	1.254	108.9
7	Acetone	15.50	10.40	7.00	0	1.756	73.8
234	O-Dichlorobenzene	19.20	6.30	3.30	1	0.671	113
617	Tetrahydrofuran (THF)	16.80	5.70	8.00	1	0.894	81.9
451	Mesitylene	18.00	0.60	0.60	1	0.990	139.5
397	Formamide	17.20	26.20	19.00	0	5.254	39.9
870	1-Chloronaphthalene	20.50	4.90	2.50	1	0.999	138.9
697	P-xylene	17.80	1.00	3.10	1	0.630	121.1
637	Toluene	18.00	1.40	2.00	1	0.681	106.6
696	Water	15.50	16.00	42.3	0	7.862	18
297	Dimethyl Formamide (DMF)	17.40	13.70	11.30	0	2.383	77.4
367	Ethylene Dichloride	18.00	7.40	4.10	0	0.725	79.4
524	Methylene Dichloride	17.00	7.30	7.10	1	0.987	64.4
325	333Ethanol	15.80	8.80	19.40	0	3.226	58.6
570	2-Propanol	15.80	6.10	16.40	0	2.560	76.9
368	Ethylene Glycol	17.00	11.00	26.00	0	4.472	55.9
698	O-Xylene	17.80	1.00	3.10	1	0.630	121.1
1078	Indene	18.70	2.60	9.00	1	0.927	116.9
333	Ethyl Benzene	17.80	0.60	1.40	1	0.724	122.8
303	Dimethyl Sulfoxide (DMSO)	18.40	16.40	10.20	0	2.168	71.3
1253	d-limonene	17.20	1.80	4.30	1	0.333	162.9
440	Isopropyl Acetate	14.90	4.50	8.20	1	0.999	117.6
430	Isobutyl Acetate	15.10	3.70	6.30	1	0.872	133.8

965	N-methyl Formamide	17.40	18.80	15.90	0	2.425	59.1
45	Amyl Acetate	15.80	3.30	6.10	1	0.694	148
328	Ethyl Acetate	15.80	5.30	7.20	1	0.714	98.6
102	n-Butyl Acetate	15.80	3.70	6.30	1	0.691	132.6
1038	1,3-Propanediol	16.80	13.50	23.20	0	2.746	72.5
500	1-Methyl Naphthalene	19.70	0.80	4.70	1	0.606	139.9
618	Tetrahydronaphthalene	19.60	2.00	2.90	1	0.547	136.7
51	Benzaldehyde	19.40	7.40	5.30	1	0.505	101.9
406	Glycerol	17.40	11.30	27.20	0	3.111	73.4
1251	Cyclopentyl Methyl Ether	16.70	4.30	4.30	1	0.433	116.5
477	Methyl Cyclohexane	16.00	0.00	1.00	1	0.996	128.2
183	Cyclohexanone	17.80	8.40	5.10	1	0.555	104.2
263	Diethylene Glycol	16.60	12.00	19.00	0	2.175	95.3
309	Dipropylene Glycol	16.50	10.60	17.70	0	2.310	131.8

In= 26 Out= 13 Total= 39
D= 17,58 P=3,73 H=4,79
Tot = 18,60
R= 6,4
Fit= 1,000
Core = ± [0,25, 1,05, 0,85]
Wrong In= 0
Wrong Out= 0

3.3 Photovoltaic Performances

Photovoltaic performances of Limonene or Benzaldehyde – processed TQ1:PC61BM active layers were investigated in polymer solar cells with a device configuration of ITO/PEDOT: PSS/TQ1:PC61BM (1:3) LiF/Al. The measurements of the polymer solar cell were carried out under illumination of AM1.5G simulated solar light at 100 mW/cm^2. The J-V characteristics are shown in fig 5 and their photovoltaic parameters are summarized in Table 4. The Limonene – processed polymer solar cell shown a maximum an open circuit voltage (Voc) of 0.7780, a short circuit current (Jsc) maximum of 9.4400, a fill factor (FF) of 51,7000 %, giving a power conversion efficiency of 3.19 %.

The Benzaldehyde – processed polymer solar cell as fig 6 and table 5 show also exhibited an open circuit voltage (Voc) of maximum 0.7580, a short circuit current (Jsc) of 5.4700, a fill factor (FF) of 42.6000 %. The Benzaldehyde processed polymer solar displayed a much lower PCE of 1.6300 %. The results show that the Limonene processed polymer solar cell is relatively better effective than the Benzaldehyde-processed solar cells even if both showed lower power conversion efficiency.

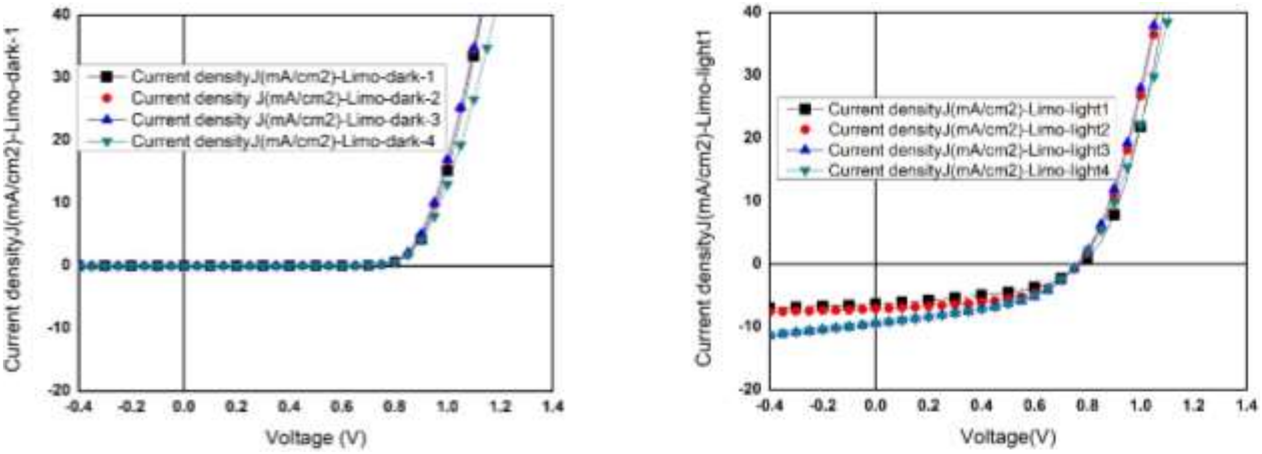

Figure 5 J-V characteristics for Limonene processed TQ1-PC61BM active layers in polymer solar cells

Table 4 Photovoltaic performances of the Limonene-processed BHJ films

No	Device	Voc [V]	Jsc[mA/cm2]	FF (%)	PCE (%)	
1	TQ1-PC61BM, Limo(1to3)	0.7780	6.3000	46.2000	2.2600	
2	TQ1-PC61BM, Limo(1to3)	0.7680	7.1300	51.7000	2.8300	
3	TQ1-PC61BM, Limo(1to3)	0.7620	9.4400	44.2000	3.1900	
4	TQ1-PC61BM, Limo(1to3)	0.7590	9.4000	44.0000	3.1400	

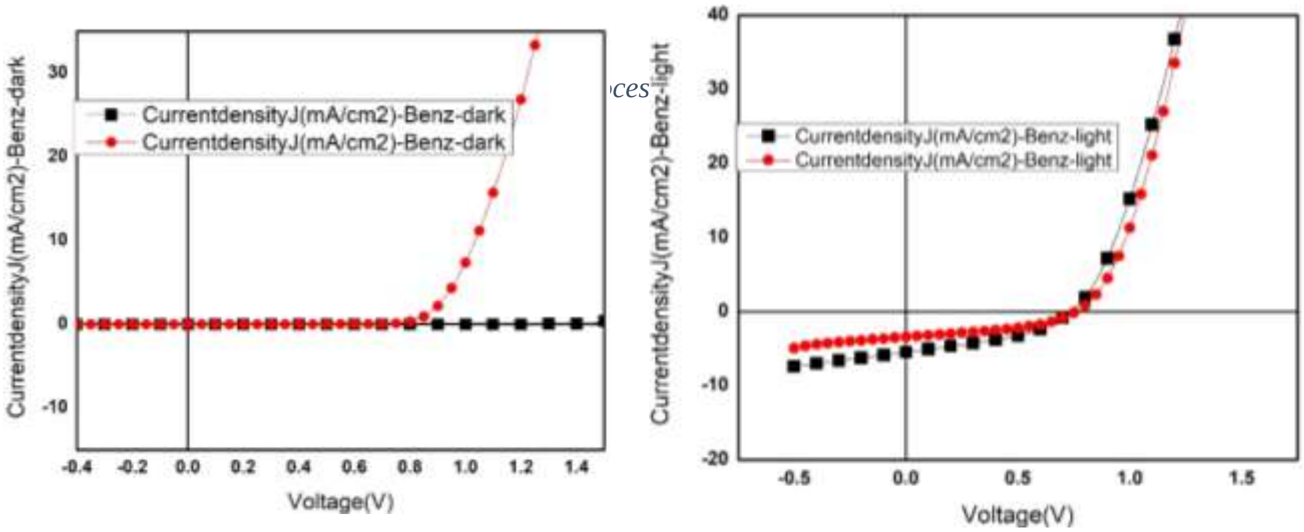

Table
5 **Phot**

Figure 6 J-V characteristics for Benzaldehyde processed TQ1-PC61BM active layers in polymer solar cells

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Table 5 Photovoltaic performances of the Benzaldehyde processed BHJ films

No	Device	Voc [V]	Jsc[mA/cm2]	FF (%)	PCE (%)
1	TQ1-PC61BM, Bez(1to3)	0.7350	5.4700	40.6000	1.6300
2	TQ1- PC61BM, Bez(1to3)	0.7580	3.4100	42.6000	1.1000

4. CONCLUSIONS

The solubility of different organic semiconductors in various solvents were determined by Hansen solubility parameters. In this case, the substituted fullerene (PCBM) and TQ1 were chosen. The Hansen Solubility parameters for both [6,6]-phenyl-C_{61}-butyric acid methyl ester ($PC_{60}BM$) and TQ1 were determined using HSPiP software. We observed a good quality of fitting parameters for PC61BM and TQ1. Therefore, Hansen solubility parameters can be taken for describing and predicting the solubility behavior of organic semiconductors.

The Hansen solubility parameters of δD, δP, δH, of Limonene were obtained from HSPiP software package and the calculation of Ra and RED of Limonene to PC61BM and TQ1 suggested that Limonene could be a good green solvent for solution processing of fullerene-based polymer solar cells. Our results suggest that Limonene would have potential to be used as green solvent for polymer solar cells fabrication if the power conversion efficiency is more increased by using suitable solvent additives.

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