

## Research Article

## Structural and Antimicrobial Investigations of Some New Silicon (IV) Complexes with Azomethines of Amino Acids

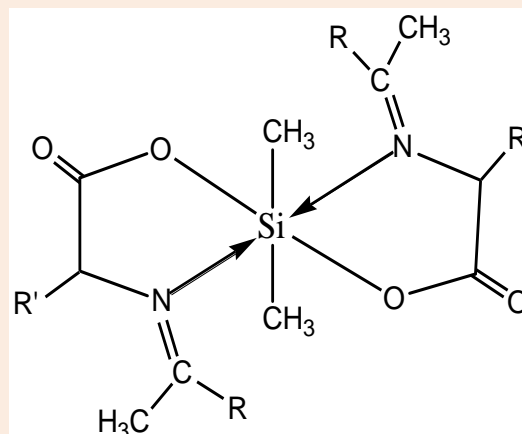
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**Abstract**

Some new silicon (IV) complexes from azomethines of amino acids have been synthesized by conventional thermal as well as microwave methods. The purity of these complexes has been checked by thin layer chromatography. The monomeric nature of these complexes has been decided by their molecular weight determination. The bonding pattern and probable geometry of these complexes have been investigated on the basis of elemental analysis, molar conductance and UV, IR, (<sup>1</sup>H, <sup>13</sup>C, <sup>29</sup>Si) NMR spectral studies. The comparative antimicrobial studies of azomethines and their corresponding silicon complexes have also been carried out and found that the complexes are superior to the free ligands and their toxicity increases by increasing concentration.

**Keywords:** Azomethines, Amino acids, Silicon (IV) complexes, Antimicrobial studies, Spectral studies

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**Introduction**

A number of ligands such as amino acids have been found to show increased biological activity after coordination with the metal ions [1-3]. Most studies have been concerned with the synthesis and characterization of their chelates with metal ions [4-7]. In recent years Schiff base complexes of amino acids have received considerable attention because of their biological activities [8-13]. The development in the field of bioinorganic chemistry has increase the interest in Schiff base complexes of amino acids, since it has been recognized that many of these compounds may serve as models for biologically important species. It have also been reported that metal complexes of amino acid Schiff bases possess anti-carcinogenic activities [14]. The present paper is a humble effort in the direction of accomplishing the systematic studies on coordination compounds of silicon with azomethines of amino acids on account of their biochemical significance. The synthesized azomethine ligands have been classified as monofunctional bidentate ligands towards the metal atom. These ligands formed a five membered chelate ring through the azomethine nitrogen atom and carboxylate oxygen.

**Experimental****Materials**

Alanine, valine, isoleucine, tryptophane, 2-acetylfluorene and 4-acetylbiphenyl were used for the preparation of Schiff base ligands. Diethoxydimethylsilane was used as starting material of silicon. The starting material and solvents used in this work were of analytical grade and were dried and purified by standard procedure. Silicon was

estimated gravimetrically as SiO<sub>2</sub>. Nitrogen was estimated by kjeldahl's methods [15-17]. Molecular weight determinations were carried out by the Rast Camphor Method.

### Physical measurement

The electronic spectra were recorded in methanol on a Toshniwal spectrophotometer. Infrared spectra were recorded on a Perkin-Elmer RX1 FTIR spectrophotometer in the region 4000–200cm<sup>-1</sup>. <sup>1</sup>H NMR was recorded on a Jeol (model FX 90Q) using DMSO-d<sub>6</sub> as solvent. <sup>13</sup>C NMR was recorded on a 90MHz Jeol (FX 90Q) NMR spectrometer using dry DMSO as the solvent at 84.25MHz. TMS was used as internal reference for <sup>1</sup>H NMR and <sup>13</sup>C NMR.

### Synthesis of ligands

The Schiff base ligands were synthesized by the condensation of 2-acetylfluorene and 4-acetylbiphenyl with amino acids (alanine, valine, isoleucine and tryptophan) in 1:1 molar ratio as previously published method [18] using methanol as the reaction medium. The solutions were refluxed on water bath for 2-3 hrs and then allow cooling at room temperature. The solid products were obtained and excess solvent was removed. It was dried further and then recrystallized from same solvent. The physical and analytical data of the ligands were recorded in **Table 1**.

**Table 1** Analytical and physical data of monofunctional bidentate ligands

S. No.	Ligand	Color	Melting Point °C	Analysis found (calcd.)%			Mol. Wt. found (calcd)
				C	H	N	
i.	2-Acetylfluorenealanine C <sub>18</sub> H <sub>17</sub> NO <sub>2</sub> (L <sup>1</sup> H)	White shining	134	76.48 (77.40)	5.72 (6.13)	4.56 (5.01)	276.22 (279.35)
ii.	2-Acetylfluorenevaline C <sub>20</sub> H <sub>21</sub> NO <sub>2</sub> (L <sup>2</sup> H)	Off white	140	77.18 (78.14)	5.38 (6.89)	4.03 (4.56)	307.00 (307.40)
iii.	2-Acetylfluoreneisoleucine C <sub>21</sub> H <sub>23</sub> NO <sub>2</sub> (L <sup>3</sup> H)	Cream	170	77.64 (78.47)	6.42 (7.21)	3.96 (4.36)	319.30 (321.43)
iv.	2-Acetylfluorenetryptophan C <sub>26</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub> (L <sup>4</sup> H)	Milky white	230	77.83 (79.17)	4.57 (5.62)	7.16 (8.11)	393.54 (394.48)
v.	4-Acetylbiphenylalanine C <sub>17</sub> H <sub>17</sub> NO <sub>2</sub> (L <sup>5</sup> H)	Off white	142	75.74 (76.38)	5.37 (6.41)	4.69 (5.24)	266.83 (267.33)
vi.	4-Acetylbiphenylvaline C <sub>19</sub> H <sub>21</sub> NO <sub>2</sub> (L <sup>6</sup> H)	Cream	154	75.95 (77.25)	7.00 (7.17)	3.79 (4.74)	294.18 (295.38)
vii.	4-Acetylbiphenylisoleucine C <sub>20</sub> H <sub>23</sub> NO <sub>2</sub> (L <sup>7</sup> H)	Pale yellow	210	77.05 (77.64)	6.94 (7.49)	3.68 (4.53)	308.02 (309.41)
viii.	4-Acetylbiphenyltryptophan C <sub>25</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub> (L <sup>8</sup> H)	Dull off white	244	77.43 (78.51)	5.26 (5.62)	6.67 (7.32)	381.73 (382.46)

### Synthesis of Complexes

#### Conventional Method

Silicon (IV) complexes were synthesized by the reaction of diethoxydimethylsilane with ligands of amino acids in 1:2 molar ratios using dry methanol as reaction medium. The contents were refluxed on fractionating column for 5-7 h. The solid product so obtained was filtered off and dried in vacuum after repeated washing with dry cyclohexane. The

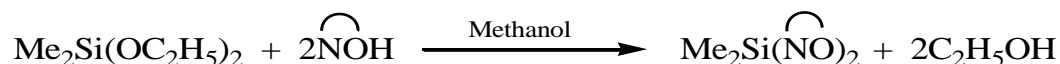
purity of compounds was checked by thin layer chromatography (TLC) on silica gel-G. The physical and analytical data of silicon (IV) complexes were recorded in **Table 2**.

**Table 2** Analytical and physical data of the silicon (IV) complexes

Reactant		Molar ratio	Product	Color	M.P. °C	Analysis found (calcd.)%			Mol. Wt. found (calcd)
Metal	Ligand					Si	N	C	
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>18</sub> H <sub>17</sub> N O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>18</sub> - H <sub>16</sub> NO <sub>2</sub> ) <sub>2</sub>	Metallic yellow solid	102	4.25 (4.57)	4.40 (4.56)	74.02 (74.24)	613.60 (614.80)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>20</sub> - H <sub>21</sub> NO <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>20</sub> - H <sub>20</sub> NO <sub>2</sub> ) <sub>2</sub>	Yellow solid	116	4.28 (4.34)	4.22 (4.33)	73.86 (74.27)	645.12 (646.89)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C. 21H <sub>23</sub> NO 2	1:2	Me <sub>2</sub> Si (C <sub>21</sub> - H <sub>22</sub> NO <sub>2</sub> ) <sub>2</sub>	Pale yellow solid	124	4.54 (4.02)	4.32 (4.01)	76.12 (75.61)	699.84 (698.96)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C. 26H <sub>22</sub> N <sub>2</sub> O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>26</sub> - H <sub>21</sub> N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub>	Yellow solid	138	3.01 (3.32)	6.04 (6.63)	76.68 (76.75)	844.78 (845.07)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>17</sub> H <sub>17</sub> N O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>17</sub> - H <sub>16</sub> NO <sub>2</sub> ) <sub>2</sub>	Orange solid	96	4.48 (4.75)	4.88 (4.74)	72.86 (73.19)	588.64 (590.78)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>19</sub> H <sub>21</sub> N O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>19</sub> H <sub>21</sub> NO <sub>2</sub> ) <sub>2</sub>	Yellow solid	122	3.62 (4.33)	3.89 (4.32)	72.47 (74.04)	644.86 (648.90)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>20</sub> H <sub>23</sub> N O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>20</sub> - H <sub>22</sub> NO <sub>2</sub> ) <sub>2</sub>	Pale yellow solid	134	4.03 (4.16)	3.94 (4.15)	74.16 (74.74)	673.97 (674.94)
Me <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	C <sub>25</sub> H <sub>22</sub> N 2O <sub>2</sub>	1:2	Me <sub>2</sub> Si (C <sub>25</sub> H <sub>21</sub> N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub>	Light yellow solid	142	3.78 (3.42)	7.04 (6.82)	77.32 (76.07)	823.25 (821.05)

### Microwave Method

A mixture of diethoxydimethylsilane and ligands of amino acid were taken in a beaker in 1:2 ratios. Minimum amount of methanol (3-5ml.) was used as reaction medium. The reaction was irradiated inside a microwave oven approx. 4-6 min. After completion of reaction the product was washed and dried under reduced pressure. Due to the rapid heating in oven through microwaves a drastic reduction in reaction time was observed. According to this procedure all the compounds were prepared in microwave.



### Biological studies

#### Antibacterial screening

All the synthesized ligands and their metal complexes were screened for in vitro inhibitory activity against the gram positive bacteria *S. aureus* and gram negative bacteria *E. coli*. These activities were evaluated by inhibition zone technique using paper disk diffusion method [19].

#### Antifungal screening

The newly synthesized silicon (IV) complexes and their ligand were screened for their activities against pathogenic fungi. The organisms used in the present investigation were *Fusarium oxysporum* and *Aspergillus flavus*. Potato dextrose agar medium (PDA) was used as test medium. The sample were prepared in different concentration and

incubated for 4-6 days at  $28 \pm 2^\circ \text{C}$ . The fungicidal screening was evaluated by radial growth method. The % inhibition was calculated by using the formula:

$$\% \text{Inhibition} = (C-T) \times 100/C$$

Where, C = diameter of fungus colony in the control plate after 4-6 days

T = diameter of the fungus colony in the tested plate after the same incubation period

## Results and Discussion

### Electronic spectra

The electronic spectra of all the synthesized ligands and their corresponding complexes were recorded in methanol solution. They shows various bands in the range of 210-225 and 250-265nm due to the charge transfer of  $\pi-\pi^*$  transitions of benzenoid and carboxylic groups, respectively. A band which was found in ligand (~342nm) due to azomethine group ( $>\text{C}=\text{N}$ ) is shifted to lower wavelength (~334nm) in complexes. So it is clearly indicate that the azomethine nitrogen participate in coordination and form a bond with silicon atom. The other bands which were found in ligand remain unchanged in complexes.

### IR spectra

The IR spectra give enough information to elucidate the nature of bonding of the ligand to the metal ions. The starting material of ligand i.e. amino acids shows strong absorption peaks at  $3370\text{cm}^{-1}$  and  $3310\text{cm}^{-1}$  which are characteristic peaks of  $\text{NH}_2$  group. In the ligand of amino acid Schiff base new sharp and strong peaks can be observed at  $1628\text{cm}^{-1}$  which is assigned for azomethine group ( $>\text{C}=\text{N}$ ) and those peaks which are observed in starting material due to  $\text{NH}_2$  stretching mode completely disappeared thus it is the clarification of forming of azomethine compound. Now on the comparison of IR spectra of ligand with their corresponding silicon (IV) complexes showed a major shift to lower wave numbers in azomethine group at  $1608-1613\text{cm}^{-1}$  it is suggesting the coordination of azomethine nitrogen with the silicon (IV) ion. Formation of silicon nitrogen bond was also confirmed by the presence of new band at  $575-550\text{cm}^{-1}$ . The IR spectra of these derivatives do not show any band assigned due to the carboxylic group. This is clearly indicated the deprotonation of carboxylic proton in the complexation and formation of oxygen-silicon bond in these complexes. It is further confirmed by the appearance of sharp band at  $518-532\text{cm}^{-1}$  in the spectra of all complexes denoted to the  $\nu(\text{Si-O})$  stretching vibrations. Thus it is conform from the IR spectra that azomethine nitrogen and carboxylate oxygen groups are involved in coordination with silicon (IV) ion in the complexes.

### $^{13}\text{C}$ NMR spectra

The  $^{13}\text{C}$  NMR spectra of ligand and their corresponding silicon (IV) complexes have been recorded. The signal due to the carbon atom attached to the azomethine group in the ligands appears at  $\delta$  164.7-166.8ppm. However, in the relative complexes the signal appears at  $\delta$  158.4-161.7 which indicate the coordination of azomethine nitrogen with the central silicon atom. Other signals which appear due to the carboxylic group at  $\delta$  177.8-182.6ppm also shifted at  $\delta$  181.8-186.3ppm and the shifts in the position indicated the participation of carboxylic group in bond formation with the silicon atom.

### $^1\text{H}$ NMR spectra

The spectrum is recorded to confirm the binding sites during the complexation. The chemical shift observed for the  $-\text{OH}$  protons in ligands ( $\delta$  11.46-11.77ppm) was not observed in any of the complexes, which confirms the bonding of the oxygen to the silicon (IV) ions ( $\text{C-O-Si}$ ). The same result was confirmed by the IR spectroscopy. The spectra of the complexes showed a singlet in the region  $\delta$  1.01-1.12ppm, which has been assigned due to  $-\text{C}(\text{CH}_3)=\text{N}-$ . The position of that signal in complexes is downfield with comparison to free ligand, suggesting deshielding due to the formation of the coordination bond between the silicon (IV) ion and azomethine nitrogen atom. The ligand shows

complex multiplet signal in the region  $\delta$  6.80-8.25ppm for the aromatic protons and these values are remains almost same position in the spectra of metal complexes. The results are given in **Table 3**.

**Table 3**  $^1\text{H}$  NMR spectral data of the ligands and their corresponding silicon (IV) complexes

Compound	Chemical shift in ( $\delta$ ppm)								
	COOH	N-CH- C	-CH- CH <sub>3</sub> / -CH <sub>2</sub> - CH <sub>3</sub>	-CH <sub>2</sub> -	-	C(CH <sub>3</sub> )=N- CH <sub>3</sub> / -CH <sub>2</sub> - CH <sub>3</sub>	CH <sub>3</sub> -CH- CH <sub>3</sub> / -CH <sub>2</sub> -C=C-	-	Aromatic protons
L <sup>1</sup> H	11.68(s)	4.48(q)	-	3.96(s)	1.09(s)	1.48(d)	-	-	6.88-8.20(m)
Me <sub>2</sub> Si(L <sup>1</sup> ) <sub>2</sub>	-	4.46(q)	-	3.98(s)	1.14(s)	1.44(d)	-	-	6.90-8.15(m)
L <sup>2</sup> H	11.54(s)	4.10(d)	-	3.92(s)	1.04(s)	1.07(d)	2.39(m)	-	6.95-7.15(m)
Me <sub>2</sub> Si(L <sup>2</sup> ) <sub>2</sub>	-	4.08(d)	-	3.89(s)	1.08(s)	1.09(d)	2.42(m)	-	7.10-7.34(m)
L <sup>3</sup> H	11.46(s)	4.02(t)	1.25(m)	3.93(s)	1.07(s)	1.12(t), 0.89(t)	-	-	7.12-7.48(m)
Me <sub>2</sub> Si(L <sup>3</sup> ) <sub>2</sub>	-	4.06(t)	1.22(m)	3.94(s)	1.12(s)	1.09(t) 0.92(t)	-	-	7.16-7.36(m)
L <sup>4</sup> H	11.72(s)	4.42(t)	-	3.87(s)	1.02(s)	-	3.23(d)	-	6.80-8.14(m)
Me <sub>2</sub> Si(L <sup>4</sup> ) <sub>2</sub>	-	4.39(t)	-	3.85(s)	1.10(s)	-	3.20(d)	-	6.86-8.12(m)
L <sup>5</sup> H	11.65(s)	4.53(q)	-	-	1.08(s)	1.47(d)	-	-	6.95-8.25(m)
Me <sub>2</sub> Si(L <sup>5</sup> ) <sub>2</sub>	-	4.56(q)	-	-	1.13(s)	1.43(d)	-	-	6.89-8.12(m)
L <sup>6</sup> H	11.59(s)	4.12(d)	-	-	1.01(s)	1.07(d)	2.38(m)	-	6.95-8.20(m)
Me <sub>2</sub> Si(L <sup>6</sup> ) <sub>2</sub>	-	4.20(d)	-	-	1.06(s)	1.04(d)	2.37(m)	-	7.04-8.14(m)
L <sup>7</sup> H	11.52(s)	3.96(t)	2.1(m)	-	1.00(s)	1.05(t) 0.88(t)	-	-	7.12-7.68(m)
Me <sub>2</sub> Si(L <sup>7</sup> ) <sub>2</sub>	-	3.99(t)	2.2(m)	-	1.07(s)	1.07(t) 0.90(t)	-	-	7.10-7.65(m)
L <sup>8</sup> H	11.77(s)	4.32(t)	-	-	1.03(s)	-	2.98(d)	-	6.98-7.84(m)
Me <sub>2</sub> Si(L <sup>8</sup> ) <sub>2</sub>	-	4.33(t)	-	-	1.12(s)	-	2.97(d)	-	7.08-7.88(m)

s = singlet, d = doublet, t = triplet, and m= multiplet.

### $^{29}\text{Si}$ NMR spectra

The value of  $\delta$   $^{29}\text{Si}$  spectra expresses the coordination number of the nucleus in the related metal complexes. Silicon (IV) complexes give sharp signals at -109.56 to -111.12ppm, which is clearly indicates the hexa-coordinated environment around the silicon atom. Thus on the basis of the above evidences it is suggested that the geometry of the resulting metal complexes can be characterized as octahedral.

### Antimicrobial results

All the free ligands and their metal complexes were screened against various bacteria and fungi to access their potential as antimicrobial agent. The data in Table 4 indicate that the Schiff base ligand L<sup>8</sup>H and their corresponding silicon complex have profound antibacterial activity against the two species; namely *S. aureus* and *E. coli*. It is noted that the presence of aromatic group increase its inhibitory effect on one or more type of bacteria as compare to alkyl group in the same position. It is also found that the gram (+) bacteria are more effected than the gram (-) bacteria. The antifungal data which is shows in Table 5 indicate that the ligands and their silicon complexes show approx. 50% inhibition against *A. flavus* and *F. oxysporum*. It is also found that the activity increased with increasing the concentration. On the basis of these results it is clearly noted that all metal complexes are more potent in their inhibition properties than the free ligands and this is in accordance with the fact that the coordination increases the activity.

**Table 4** Antibacterial activity data of ligands and their corresponding silicon (IV) complexes:-  
Inhibition zone in mm after 24h at 28<sup>o</sup> C (conc. in ppm)

Compound	Diameter of inhibition zone (mm)			
	<i>E. coli</i> (-)		<i>S. aureus</i> (+)	
	500ppm	1000ppm	500ppm	1000ppm
C <sub>18</sub> H <sub>17</sub> NO <sub>2</sub> [L <sup>1</sup> H]	4	6	6	7
Me <sub>2</sub> Si(C <sub>18</sub> H <sub>17</sub> NO <sub>2</sub> ) <sub>2</sub>	6	8	7	9
C <sub>21</sub> H <sub>23</sub> NO <sub>2</sub> [L <sup>3</sup> H]	5	7	6	8
Me <sub>2</sub> Si (C <sub>21</sub> H <sub>22</sub> NO <sub>2</sub> ) <sub>2</sub>	8	10	9	10
C <sub>17</sub> H <sub>17</sub> NO <sub>2</sub> [L <sup>5</sup> H]	6	8	7	9
Me <sub>2</sub> Si(C <sub>17</sub> H <sub>16</sub> NO <sub>2</sub> ) <sub>2</sub>	8	9	8	10
C <sub>25</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub> [L <sup>8</sup> H]	7	8	8	10
Me <sub>2</sub> Si(C <sub>25</sub> H <sub>21</sub> N <sub>2</sub> O <sub>2</sub> ) <sub>2</sub>	10	12	11	13
Streptomycin	15	18	16	19

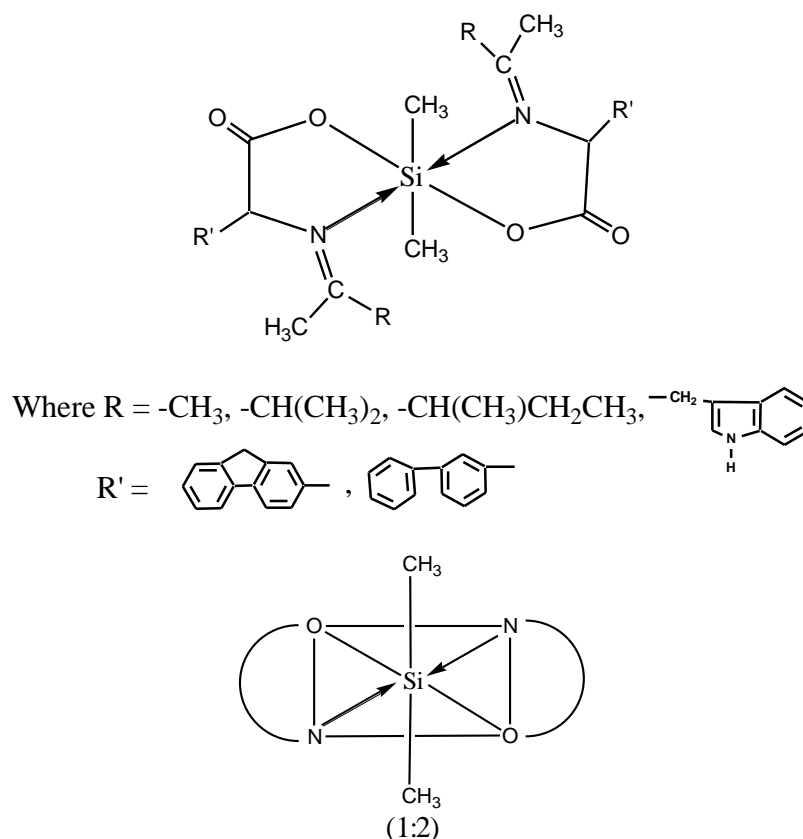
**Table 5** Antifungal activity data of ligands and their corresponding silicon (IV) complexes:-  
Inhibition (%) after 96h at 28<sup>o</sup> C (conc. in ppm)

Compound	Diameter of inhibition zone (mm)					
	<i>F. oxysporum</i>			<i>A. flavus</i>		
	50 ppm	100ppm	200 ppm	50 ppm	100 ppm	200 ppm
C <sub>18</sub> H <sub>17</sub> NO <sub>2</sub>	26	38	44	30	38	50
Me <sub>2</sub> Si(C <sub>18</sub> H <sub>17</sub> NO <sub>2</sub> ) <sub>2</sub>	28	40	50	33	42	56
C <sub>21</sub> H <sub>23</sub> NO <sub>2</sub>	23	35	46	27	39	51

$\text{Me}_2\text{Si}(\text{C}_{21}\text{H}_{22}\text{NO}_2)_2$	33	43	54	36	45	60
$\text{C}_{17}\text{H}_{17}\text{NO}_2$	25	37	45	28	39	48
$\text{Me}_2\text{Si}(\text{C}_{17}\text{H}_{16}\text{NO}_2)_2$	29	41	49	33	42	56
$\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_2$	24	39	44	24	39	50
$\text{Me}_2\text{Si}(\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2)_2$	30	45	50	34	46	60
Micostatin	70	81	94	72	84	96

## Conclusion

Microwave synthesis gives a very economic and ecofriendly platform to synthesize these type of complexes with good yield of products. Spectroscopic studies suggested that the Schiff base ligands of amino acids coordinate to the silicon (IV) ion in bidentate manner so that such complexes shows octahedral geometry having the composition  $\text{Me}_2\text{ML}_2$ . The IR spectral studied shows the deprotonation of carboxylic group, coordination of carboxylic oxygen and participation of nitrogen of azomethine group in complexation. These results also supported by NMR studies. The antimicrobial results shows that the complexes having more potential than the corresponding ligands. The structure of resulting complexes can be represented as given below. (Figure 1)



**Figure 1** Structure of the silicon (IV) complexes

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