



## Synthesis and experimental analysis of polydimethylsiloxane (PDMS) based substrate with cobalt (green synthesized) nano composite material

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### Abstract

The world is entering in new era where miniature technology is becoming the focal point of fast progress, to fulfill and complete the modern-days requirements. The need to cope with rapid and complex challenges in this concern to report is first endeavor to achieve the worthwhile outcomes. Poly (dimethyl) Siloxane, a silicon based elastomer having two methyl group (thus the name comprises -di in its name) which is a thick viscous liquid is solidified having various patterns on its surface; the produced patterns enhance the adhesion power of the solidified PDMS. Following this, experiments includes preparation of cobalt nanoparticles using chemical reduction method via cobalt nitrate as the precursor, the method utilizes methanol as a capping agent, preventing the oxidation as well as agglomeration problem once the particles have been prepared successfully. Finally, the characterization techniques had been performed like SEM, XRD technique which confirmed the formation of nanoparticles leads to PDMS based composite. Experiments depicts that extremely thin composite layers of PDMS substrates having very fundamental nature of polymeric class of materials, i.e. dissolution and disintegration property, which can be utilized in medical, electronic and other useful industries in the coming days.

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## Introduction

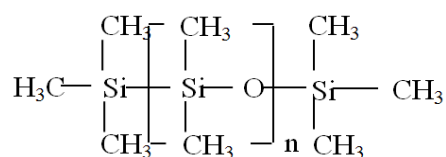
Human epidermis is the outer region of two layers of cells which make human skin & inner one being the dermis. The human skin is the structure that consist these separate cells forming layers, layers of skin and tissue on the human muscle, which acts as a protecting layer against destruction from environmental pathogens and temperature changes (Mehdi *et al.*, 2015). This is the largest and one of the most interesting organs of human body. As the skin is a stretchable & flexible structure, therefore mechanical behavior of the skin has been studied for long time in assessments being as part of development for cosmetic & clinical uses (El-Molla *et al.*, 2016). Especially studies of the mechanics of epidermis is important since it's the most upper skin layer that interfaces with the products of cosmetics & where skin diseases effects firstly. Moreover, mechanical behavior of epidermis is strongly effected by environment such as temperature, weather & humidity. In a vivo condition, epidermis is also hydrated by under layer of dermis, thus there is need for study on mechanical behavior of different skin layers (Wang *et al.*, 2014).

For these reasons, it had been utmost investigation by reducing the modulus and thickness of PDMS which makes it comparative with properties of human epidermis & give these results useful for the epidermal electronics as well. Moreover, they can be used in clinical uses for doing skin surgeries to remove or to help in regrown process of dead skin cells, which had been damaged or destroyed by fires, electrical shocks or some different kind of accidents (Mehdi *et al.*, 2015).

This research shall provide a mode of initiation for studying different effects of variation of CONP (Cobalt Nanoparticles) coated with PDMS substrates to form structure with similar composition, further involvement in fields of bio medical applications, stretchable electronics, solar cells, smart textiles etc (Rösch *et al.* 2000). For achieving these objectives, soft substrate is to be needed which contains as many elastomers like silicon based elastomers that have

versatile properties as biocompatibility, flexibility, non-toxicity, hydrophobicity, stretch ability etc. They should have coated with appropriate thin films of metals in order to conduct electricity and transmit heat. Polydimethylsiloxane (PDMS) has taken as soft elastomeric substrate in this study because of its versatile properties such as bend ability, stretch ability and biocompatibility (Agar, 2011). Important aspect of study is to fabricate an elastic layers of PDMS that can bear applied strain incomparison of skin and synthesized cobalt nanoparticles coated which acts as a conducting thin film on PDMS substrate.

PDMS is a polymeric organo-silicon product which is generally said to be silicones. It is most utilized silicon based product and important for its relatively flowing properties. P.D.M.S is chemically inert, transparent, non-flammable & non-toxic. Generally, it is available in a cleaned room type of silicone rubber with many useful applications. On observing the formula in Fig 1. it is proved that the methyl functional group has an incredible effect of its properties. Due to flexible polymeric backbones and chains, PDMS molecules have strong siloxane linkages. So, they are loosely bonded when the molecular weight is high that results in P.D.M.S unusual high level of viscos-elasticity.



**Fig. 1.** Structure of PDMS, two methyl functional groups are mainly responsible for unique properties. (Lötters JC, Olthuis W, Veltink PH, Bergveld P. 1997. Mills KL, Zhu X, Takayama S, Thouless MD. 2008).

PDMS is a clear elastomer, which is very common material used frequently in the applications of bioengineering, stretchable electronics, optofluidic and Microelectromechanical systems (MEMS). Specifically, some of these applications include micromachined mechanical & chemical sensors, stretchable solar cells, strain sensors, nanogenerators and microfluidics devices.

PDMS is widely used because of its biocompatibility, gas permeable, flexibility, non-toxicity, hydrophobicity, stretch ability and good for rapid prototyping of devices (Mojsiewicz-Pieńkowska *et al.*, 2016).

PDMS is a biocompatible thermally set polymer that could easily mold and modified. (Mehdi *et al.*, 2014). Objectives for this project are ,Forming Poly Di Methyl Siloxane substrates, Synthesizing cobalt Nano Particle, Characterization of cobalt nanoparticles, Formation of PDMS substrates & composite with it cobalt nanoparticles, Testing the different types of substrate samples formed & studying degree of stretch-ability, disintegration dissolution & flexibility of each substrate with respect to human epidermis & human body chemicals, Measuring mechanical properties of PDMS network coated with cobalt nanoparticles to a soft materials mechanical properties with respect to human body.

## Material and methods

### *Synthesis of Cobalt Nanoparticles (CONP's)*

All of reagents like Methanol & Cobalt Nitrate that are utilized all over work research analytically graded by Pakistan and Sigma Aldrich, Merck, United State America. All the Equipment that were used are Magnetic stirrer with Hot plate (MS-H-Pro+), Spectro-photometer (Tomos), Analytical balance (Sartorius from Germany), SEM analyzer (Hitachi S4160 of Japan), grinder (West point), the vacuum filtration of assembly (Thomas 4595D45), XRD analyzer (Karaltay, DX-2700 MIN), & Thermostat (Siemens). Samples are obtained from N.E.D University main campus Karachi & for 1 week they were dried & then grinded into small particles. 100 grams of shaded-dried leaves are converted into powder after that 500 Milliliters of methanol, ethanol & de-ionized H<sub>2</sub>O in 1 Liter flask is added & mixed gently. We can prepare plant extract by heating magnetic stirrer to 50° Celsius for about 1 hour. Vacuum filtration assembly is used to filter the total extract. CoNP's were to be prepared in 250mL of the conical flask in which the 50mL concentration of cent molar solution of the cobalt nitrate salt was mixed

with the 10mL of plant extract (100 gram of the dried leaves powder was added to 500mL methanol solution, ethanol and De-ionized water in the 1 Liter flask) with continuous shaking on the hot plate till dark brown color appeared.

### *PDMS specimens preparation*

Network of PDMS samples of this research are basically synthesized with the same composition, that are Sylgard 184 silicone elastomer curing agent and Sylgard 184 silicone elastomer base. These samples had different base & agent ratios, that mean there are different degrees for cross-linking. If the degree of the PDMS network is lower cross-linking than softer the PDMS, if the degree is higher for cross-linking the sample will be harder. Therefore, sample stiffness changes by the ratio of cross-linker to the base polymer in this thesis.

Mostly utilized type of the PDMS network in this research is PDMS 1:10, that means 1 mass of Sylgard 184 silicone elastomer curing agent with 10 mass of Sylgard 184 silicone elastomer base. For the PDMS network, there are different base and agent ratio means different amount of the cross-linking.

In this research, there is a series of the PDMS network samples with the different base/agent ratios that are being utilized to study the relationship between changing of modulus & the amount of the PDMS network's cross-linking that are different, are PDMS network 0.7:10, PDMS network 0.5:10, PDMS network 0.3:10 & PDMS network 1:10 We used ratio 1:10 and making sample and composite of 2.5, 1, 0.5 and 0.3% (W/W).

The formation of PDMS specimens requires elastomer curing agent Sylgard 184 silicone, elastomer base Sylgard 184 silicone, wood spoons, petri dishes, vacuum desiccator, beakers, scale, gloves & hot plates. The 2.5 ,1 ,0.5,0.3gm respectively for simple PDMS elastomer base Sylard 184 silicone is poured in beaker & tare generally. In the second stage, slow pouring of 0.25, 0.1, 0.05 ,0.03gm respectively of cross linker for simple PDMS & composite respectively of elastomer curing agent

Sylard 184 silicone in beaker & stir by help of pipette. Mixed it with the glass rod for 10 minutes, until the mixture turn into a milky solution. Initially the mixture was full of air bubbles so removal of air bubbles by centrifuging it with the help of glass rod. Under vacuum removal of air bubbles for (20~30 min) de-gassing.

Then P.D.M.S solution & composite transfer on an empty mold & again bubble repair so put them again in sonication for degassing and then placed in an oven for 100 degree Celsius for 3 hours and after that allow the samples to place in room temperature for few hours. The preparation stages of PDMS substrate/composite formations are completed by peeling through blade or knife from base.

**Results and discussion**

Therefore, PDMS along with its composite very highly stable product the result shows that they don't disintegrate on 0.1,1 N HCL which gives highly reflection of their flexibility, stretch ability, stability they only disintegrate on very high extreme condition of HCL by placing sample at 12 hours which showing leaching affect in PDMS.

**Table 1.** Disintegration Test of PDMS & Composite by Using 0.1 N HCL at Different Time Interval.

Sample (% w/w)	Results
At 15 Mints. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No effect)
At 30 Mints. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No effect)
At 45 Mints. (COMPOSITE+PDMS SAMPLE of 2.5,1, 0.5&0.3%)	(No effect)
At 60 Mints. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No effect)
For 6 hours (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No effect)

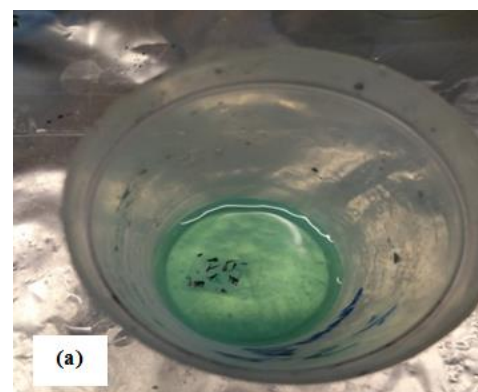
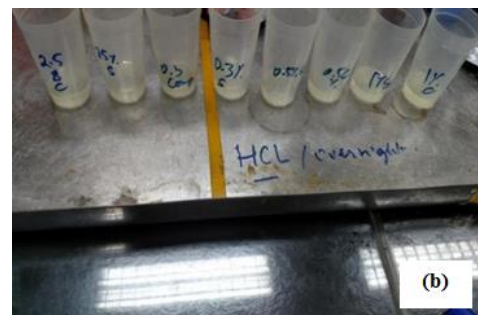
**Table 2.** Disintegration test of PDMS Composite by using 1N HCL at different time interval.

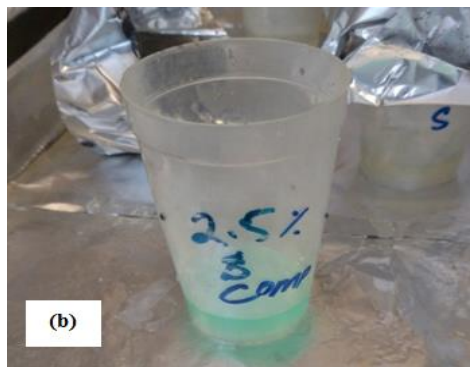
Sample (% w/w)	Results
At 15 Mint. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
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At 45 Mints. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
At 60 Mints. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
For 6 hours (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)

**Table 3.** Disintegration test of PDMS & Composite by using 37% HCL (Conc.) at different time interval.

Sample (% w/v)	Results
At 15 mint. (Composite +PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
At 30 mint. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
At 45 mint. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)
At 60 mint. (Composite + PDMS sample of 2.5,1, 0.5&0.3%)	(No Effect)

PDMS Sample & PDMS composite of 2.5, 1, 0.5 & 0.3% (w/w) also placed for 12 hours over night on next day the result are all the sample having slightly etching occurs which has been identified by the color change of cobalt composite PDMS, because when sample itched the cobalt react with concentrated HCL so then it will converted into Cobalt Chloride that's showing sea green color of sample, which placed over night as seen in attached image.





**Fig. 1.** A & B HCl containing PDMS composite (Result after 12 Hour) showing change in color (transparent to sea green) of HCl due to itching in sample because Cobalt react with HCl.

*Dissolution Test Result for Sample & PDMS Composite At Different Condition*



**Fig. 2.** Dissolution Apparatus set with the condition of normal body temperature.

*Dissolution Test Result for Sample & PDMS Composite At Different Condition*

**Table 4.** Observations extracted from U-V spectroscopy technique (Spectrum) by using 0.1N HCL as a dissolution medium.

PDMS Concentration (w/w)	Absorbance		
	Max. Wavelength (200-400)	Simple PDMS	Composite PDMS
0.3%	290	0.047	0.007
	261	0.017	NA
	231	0.097	NA
	340	NA	0.013
0.5%	366	0.007	NA
	290	0.062	NA
	230	0.108	NA
1 %	283	0.008	NA
	307	NA	0.002
2.5%	NA	NA	NA

Now the sample is testing for dissolution aspect i.e. dissolution medium prepared of 0.1 N HCl Concentration in which the sample is placed and set the 100 RPM of dissolution apparatus also set the temperature of apparatus 37 degree Celsius with respect to human body temperature and then with drawn sample after one hour from dissolution beaker and test on UV apparatus with wave length range (200-400), so we get the above mention value of absorbance of PDMS sample and PDMS composite at their maximum wave length.

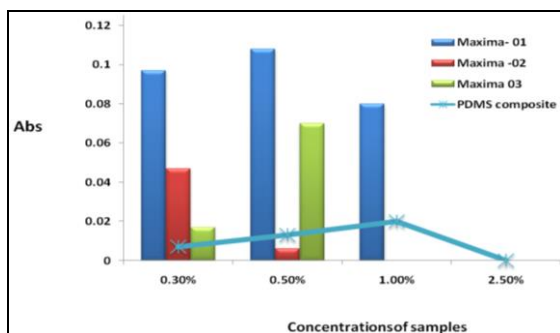
**Table 5.** Observations extracted from U-V spectroscopy technique (Spectrum) by using 1 NHCL as a dissolution medium.

PDMS Concentration (w/w)	Absorbance		
	Max. Wavelength (200-400)	Simple PDMS	Composite PDMS
0.3%	221	0.042	NA
	219	NA	0.041
	367	NA	0.007
0.5%	341	NA	0.010
	338	NA	0.014
1 %	341	NA	0.006
	307	0.015	NA
2.5%	NA	NA	NA

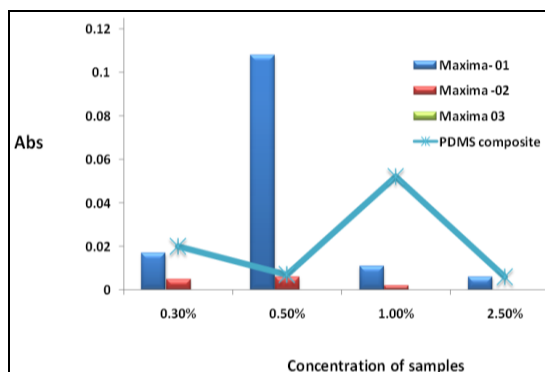
Now the sample is testing for more high concentration of dissolution medium which is prepared of 1 N HCl Concentration in which the sample is placed and set the 100 RPM of dissolution apparatus also set the temperature of apparatus 37 degree Celsius with respect to human body temperature and then with drawn sample after one hour from dissolution beaker and test on UV apparatus with wave length range (200-400), so we get the above mention value of absorbance of PDMS sample and PDMS composite at their maximum wave length. Now the sample is testing for the aspect by changing dissolution medium i.e Phosphate buffer of Ph 5.4 Concentration in which the sample is placed and set the 100 RPM of dissolution apparatus also set the temperature of apparatus 37 degree Celsius with respect to human body temperature and then with drawn sample after one hour from dissolution beaker and test on UV apparatus with wave length range (200-400), so we get the above mention value of absorbance of PDMS sample and PDMS composite at their maximum wave length.

**Table 6.** Observations extracted from U-V spectroscopy technique (Spectrum) by using phosphate buffer as a dissolution medium.

PDMS Concentration (w/w)	Absorbance		
	Max. Wavelength (200-400)	Simple PDMS	Composite PDMS
0.3%	292	0.005	NA
	269	NA	0.020
	215	0.017	NA
0.5%	366	0.006	0.007
	269	NA	0.015
	215	0.010	0.014
1 %	366	0.011	NA
	342	NA	0.051
	214	0.002	NA
2.5%	366	0.006	0.013



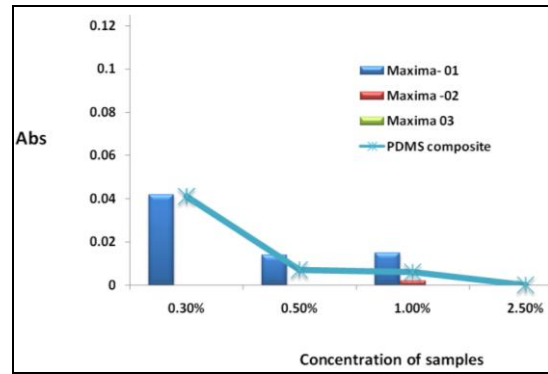
**Fig. 3.** Absorbance of different concentrations sample by using 0.1 N HCL.



**Fig. 4.** Absorbance of different concentrations sample by using 1 N HCL.

*Graphical Representation*

The bar graph shows the absorption result of simple PDMS substrate in which all the sample showing some absorption tendency except concentration of 2.5, Similarly the line graph shows the absorption result of Cobalt composite PDMS substrate in which all the sample showing some absorption tendency except concentration of 2.5 when using 0.1 N HCL as a dissolution medium.



**Fig. 5.** Absorbance of different concentrations sample by using phosphate buffer of PH 5.45.

The bar graph shows the absorption result of simple PDMS substrate in which all the sample showing some absorption tendency except concentration of 2.5, Similarly the line graph shows the absorption result of Cobalt composite PDMS substrate in which all the sample showing some absorption tendency except concentration of 2.5 when using increase concentration i.e. 1 N HCL as a dissolution medium.

**Discussion**

In the end we found the results from above graphs that by using 0.1 N HCL as a dissolution medium (condition num:1) have shown some absorption on their maximum wavelength so they have compatibility to use as a medium for medical technology similarly for condition number 2 also but in condition number 3 using phosphate buffer of Ph 5.4 is more favorable because all of their sample show some absorption properties which is quite better than condition number 1 & 2 & more accepted for some medical technology if it use as a medium because both composite & simple PDMS of condition number 3 have also mechanical stretch ability, flexibility, stability and up to some extent of absorbing properties & would use in various electrical & medical devices for sensitivity purpose.

Thus, we also conclude that PDMS is very high stable product and not disintegrate through various test of disintegration so it stability also beneficial for the skin of human and as useful for surface wearable devices. For these stretchable, thin, flexible, soft & customizable which could be directly worn on the skin are being

manufactured by chemical industries using this data. This is a vast field relating to newer technology advancements & we hope that someday somewhere our work will help become part of a newer revolution in the technology of Cobalt coated PDMS in the fields of stretchable electronics & in medical revolution.

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