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Application of Experimental Design for Electroless Nickel Plated Composite Textile Materials for Electromagnet Compatibility

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Abstract
The electroless plated nikel textile has been optimized with different factors and levels after designing the experiment. The higher the better case has been identified from the process owners. After careful analysis it is observed that there is an increase of palladium chloride and nickel (II) sulphate. This shows the increase in nickel deposition and electromagnetic shielding between 200-1000 MHz.

1. Introduction

Robust design method, also called the Taguchi method, pioneered by Dr. Genichi Taguchi, greatly improves engineering productivity. By consciously considering the noise factors (environmental variation during the product's usage, manufacturing variation, and component deterioration) and the cost of failure in the field the Robust Design method helps ensure customer satisfaction. Robust Design focuses on improving the fundamental function of the product or process, thus facilitating flexible designs and concurrent engineering. Indeed, it is the most powerful method available to reduce product cost, improve quality, and simultaneously reduce development interval.

Over the last five years many leading companies have invested heavily in the Six Sigma approach aimed at reducing waste during manufacturing and operations. These efforts have had great impact on the cost structure and hence on the bottom line of those companies. Many of them have reached the maximum potential of the traditional Six Sigma approach

2. Literature Review

Electromagnetic shielding provides protectionby reducing signals to levels at which they nolonger affect equipment or can no longer be received. Reflecting and absorbing the radiation achieve this. Polymeric composites are extensively used aspassive and active elements in some electricalcircuit components in different technological applications due to their lightweight, easy processability, flexibility, corrosion resistance andcost-effectiveness. But elastomers and plastics contain verylow concentrations of free charge carriers. Theyare electrically non-conductive and transparentto electromagnetic radiation. To provideconductivity and shielding from ElectromagneticInterference (EMI), incorporation of fillers of highintrinsic conductivity such as particulate carbon blacks,carbon andgraphitefibers,ormetalpowdertothepolymer matrix is required. The amount ofelectrically conductive filler required to impart highelectrical conductivity to an insulating polymer canbe dramatically decreased by the selective localizationof the filler in one phase, or best at the interface ofa continuous two-phase polymer blenddue to their light weight, easy processability, flexibility, corrosion resistance andcost-effectiveness. But elastomers and plastics contain verylow concentrations of free charge carriers. Theyare electrically non-conductive and transparentto electromagnetic radiation. To provideconductivity and shielding ElectromagneticInterference (EMI), incorporation of fillers of highintrinsic conductivity such as particulate carbon blacks,carbon andgraphitefibers,ormetalpowdertothepolymer matrix is required. The amount ofelectrically conductive filler required to impart highelectrical conductivity to an insulating polymer canbe dramatically decreased by the selective localizationof the filler in one phase, or best at the interface ofa continuous two-phase polymer blend

Unfortunately most polymer composites used forhousehold electrical and electronic devices areelectrically insulating and transparent to electromagneticradiation and electrostatic discharge (ESD). The potential health hazards (e.g. cancer) associated withexposure to electromagnetic fieldsare also the matterof concerned. To shield and limit against EMI andESD, conductive polymer composites startedreplacing coated materials for various shielding applicationsin the electrical and electronic industries, especiallyfor electronic household materials. This trend hasbeen driven mainly because of the better characteristicsof these polymers in terms of ESD, shielding fromEMI, thermal expansion, density, and chemical(corrosion and oxidation resistance) properties.However, conductive polymers have rigid characteristicsowing to their chemical conformation of benzenerings, making their formation difficult. A few studieshave reported conductive knitted fabricsreinforced composites as electromagnetic shieldingeffectiveness (EMSE)andESDmaterials.Previously,Cheng*etal*.fabricated some knitted fabricreinforced polypropylene composites for use as EMSE andESD materials, and indicated that the knittedfabric reinforced polymer composites are suitable for making complex shaped components andapplication in electromagneticshieldingElectroless plating is a chemical reduction process which depends upon the catalytic reduction ofa metallic ion in an aqueous solution containinga reducing agent, and the subsequent deposition ofthe metal without the use of electrical energy. Themetals capable of being deposited by electrolessplating include nickel, cobalt, copper, gold,palladium and silver.

Electroless nickel (EN) is an alloy of88-99% nickel and the balance with phosphorous, boron, ora few other possible elements. EN coatings,therefore, can be tailored to meet the specific requirements ofan applicationwiththeproperselectionofthenickel's alloying element(s) and their respectivepercentages in the plated layer..

Diligent control of the solution's stabilizercontent, *p*H, temperature, tank maintenance, loading,and freedom from contamination are essential forits reliable operation. EN solutions are highlysurface area dependent. Surface areas are introduced tothe solution by the tank itself, in-tankequipment, immersed substrates, and contaminants.Continuous filtration, often sub-micron, of the solution at a rateof atleast ten turnovers per hour is alwaysrecommended toavoidparticlecontaminationwhichcouldleadto solution decomposition or imperfections in theplated layer. In this study, attempts have been madeto optimize the electro less nickel plated process parameters to improve the EMSE of copper coreyarn with polyester sheath composite fabricthrough Taguchi design andANOVA.

3. **Schematic Representation of the Steps Involved in the Taguchi Optimization Procedure**.

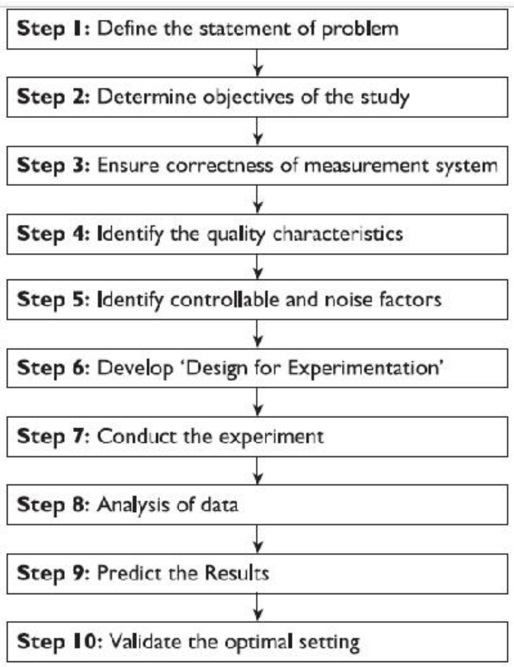

Figure-1 – Steps of optimization

4. Methodology

Step 1: Statement of the problem.

Electroless nickel plated composite textile materials for electromagnet compatibility**.**

Step 2: Objectives of Study.

- To understand the deployment of Taguchi approach for problem-solving;
- To introduce a systematic 10 step Taguchi approach;
- Pre-treatment
- Pre –cleaning
- Sensitisation
- Activation
- Deposition
- Post-treatment
- ExperimentalDesign
- Electromagnetic ShieldingEffectivenessTest
- Shielding Effectiveness
- TestProcedure
- EffectsofPalladiumChloride ConcentrationonEMSE.

Step 3: Measurement System Analysis.

3.1 Pre-treatment

To facilitate the plating of metal particles onthe fabric surface, pre-treatment was carried out.The processes of pre-treatment consist ofpre-cleaning, sensitization andactivation

3.2 Pre-cleaning

Pre-cleaning is done for operating theelectroless nickel solution so that required properties areobtained in the fabric. Additives and/or contaminants canhave a profound effect on performance, as willoperate conditions. In this process, all fabric sampleswere pre-cleaned in a 2% non-ionic detergent at *p*H 7and temperature 40°C for 20 min. Deionised waterwas then used to rinse the pre-cleaned samples. TritonX- 100 as a non-ionic detergent wasused.

3.3 Sensitisation

Sensitisationisa processofcatalyzingthe non-conductivesubstrate.Thisenablesthefabricsurfacetoactasacatalystforthedepositionofnickel.Theauto-catalysationisobtainedthroughthisprocess.Inthecaseofsensitizingfabricsurfaces,thecleanedfabricsamplesweresubjectedtothesurfacesensitizer mixtureof5g/Lstannouschlorideand5M/Lhydrochloricacid)withslowagitationfor10minat25°Cand1*p*H.Insensitization,non-conductivesubstrateabsorbsthestannousSn^{2+}ionsfromthestannoussolutions.

3.4 Activation

Activationiscarriedoutforactivatingthefabricsurfaceforthenickeldeposition.Theobjectiveofthisprocessissameasthatofsensitization.Inthisprocess,aactivatorsolutionisused.Thesensitizedfabricswererinsedwithdeionisedwatersubsequentlyandthenimmersedintheactivatorsolution[palladium

(II)chloride,hydrochloricacid(conc.37%)andboricacid]at*p*H2and25°Cfor5mininordertoachievesurfaceactivation. Theactivatedfabricswererinsedwith deionisedwaterafterwards.

Activationiscarriedoutforactivatingthefabricsurfaceforthenickeldeposition.Theobjectiveofthisprocessissameasthatofsensitization.Inthisprocess,aactivatorsolutionisused.Thesensitizedfabricswererinsedwithdeionisedwatersubsequentlyandthenimmersedintheactivatorsolutionpalladium

(II)chloride,hydrochloricacid(conc.37%)andboricacid]at*p*H2and25°Cfor5mininordertoachievesurfaceactivation. Theactivatedfabricswererinsedwith deionisedwaterafterwards.

3.5 Deposition

Depositioninvolvesreducingthenickelsaltintonickel,i.e.themetalionsarereducedasmetalsbythereducingagent.Incaseofelectroplating,electriccurrentisusedforthereductionprocess.Inthisdeposition,sodiumhypophosphitemonohydrateisusedasreducingagent.Duringthedepositionstage,thenickelplatingsolutionwasemployedintheelectrolessnickelplatingtank,whichwascomposedofnickel(II)sulphatehydrate,tri-sodiumsaltdehydrate,ammoniumchloride, a fewdropsofsodiumhydroxide(conc.10%)andsodiumhypophosphitemonohydrate.Theactivatedfabricswereimmersedinthenickelplatingsolutionat*p*H9withvarioustemperatureandtime,rightafterthemetallisingreactionofelectrolessnickelplating.

3.6 Experimental design

Taguchimethodwasappliedtoidentifytheoptimumlevelofelectrolessnickelprocessparametersforelectromagneticshieldingeffectivenessperformancecharacteristicindividually.Taguchimethodreplicateseachexperimentwiththeaidofanouterarraythatdeliberatelyincludesthesourcesofvariationthataproductwouldcomeacrosswhileinservice.Suchadesigniscalledaminimumsensitivitydesignorarobustdesign.TherobustdesignmethodiscalledTaguchimethod.
Toachievetheoptimumdesignfactorsetting,Taguchiadvocatedacombinationoftwostageprocess.Firststepisrelatedtotheselectionofrobustnessseekingfactorsandthesecondstepisrelatedtotheselectionofadjustmentfactorstoachievethedesiredtargetperformance.Variousstagesintheexperimentaldesignhavebeendealtbymanyresearchersinthepast.Inotherexperimentaldesigns,uncontrollablefactorsarekeptunderobservationduringexperimentation,whereasTaguchi

3.7 Degreesof freedom(DOF)of OA≥TotalDOFrequired Therefore,Taguchiorthogonalarrayand 3levelswereselectedtoassignvariouscolumns.The

Table 1— Signal-to-noise (S/N)ratioandits significance

Case	S/N ratio
Target is the best	S/N () = 10log10($_2$n^2/s^2)
Small-the-better	S/N () = -10log10(yi/ n)
Larger-the-better	S/N () = 10log10[(1/yi^2) / n]Binaryscale (GO/NO-GO) S/N () = 10log10(p/1-p)

3.8 Electromagnetic ShieldingEffectivenessTest

TheEMSEoftheconductivefabricwascalculatedbyfollowingASTMD4935–99testmethods.Thebasiccharacteristicoftheconductivefabricisitsattenuationproperty.Attenuationoftheelectromagneticenergyisaresultofthereflection,absorptionandmulti-reflectionlossescausedbyaspecificmaterialinsertedbetweenthesourceandthereceptoroftheradiatedelectromagneticenergy.Attenuationcausedbyamaterialischaracterized,dependingonthemeasuringmethodused,bythetwoquantities,namelyshieldingeffectiveness(SE)andinsertionloss(A)

3.9 Shielding Effectiveness

Shelding effectiveness (SE) is defined as ratio of electromagnetic strength(E_0)measuredwithandwithoutthetestedmaterial(E_1)whenitseparatesthefieldsourceandthereceptor, this depends on the difference between source and receptor of electromagnetic energy. In the far field zone its characteristics the attenuation of electromagnetic wave the mearsument s are carried out inthenearfieldzonecharacterizestheattenuationeffectivenessfortheelectricormagneticfieldcomponentonly

Step 4: Identify the characteristics

Thetestswerecarriedoutinananechoicchamber.Anechoicchamberisaroominwhichnoacousticalreflectionsorechoesexist.Thefloor,wallsandceilingsoftheseroomsarelinedwithametallicsubstancetopreventthepassageofelectromagneticwaves.Radiofrequencysignalwasgeneratedbyasignalgeneratoranditwastransmittedthroughanantennaoutsidethechamber.Signalfromthesignalgeneratorwasmeasured
byspectrumanalyzerwithantennainsidethechamber.Thefirstmeasurement(calibration)wascarriedoutwithoutthetestfabric.Thetestsinthedifferentfrequencyrangeswereconducted

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Step 5:Identify controllable factors and factor levels

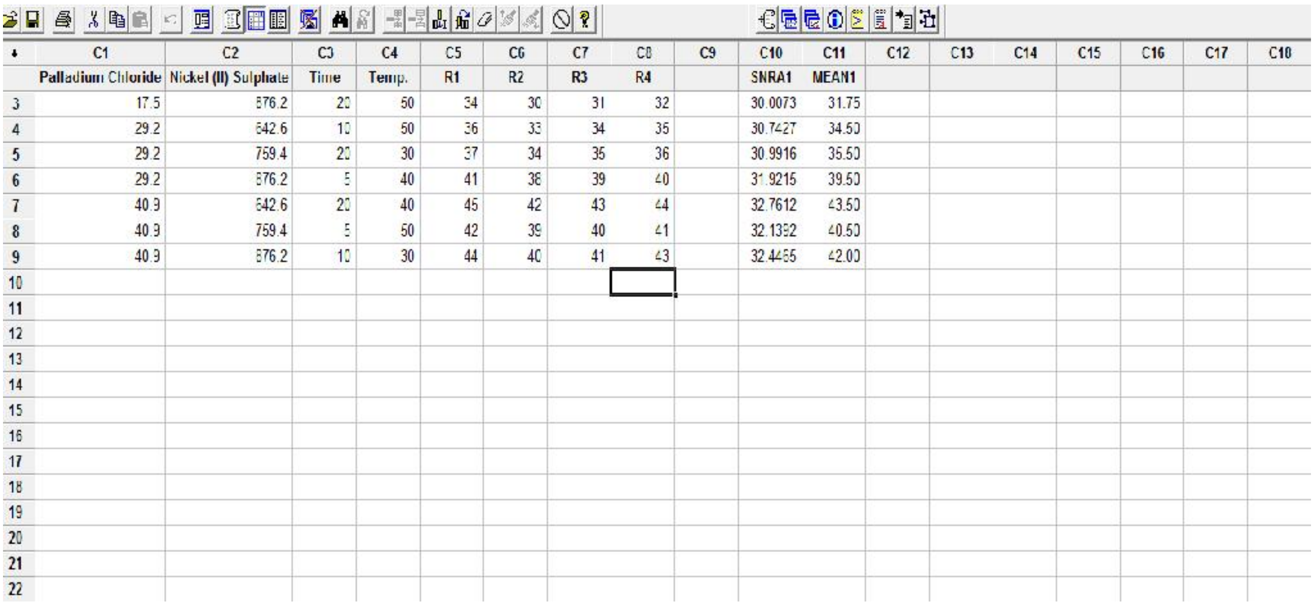
Table-1 Factors and Levels

Factors	Levels		
A	17.5	29.2	**40.9**
B	642.6	759.4	**876.2**
C	5	10	**20**
D	**30**	**40**	**50**

Step 6:Development of experimental design

After collecting information about the factors and factor levels, the experimental design is prepared. The experimental design was prepared by considering

Experimentaltestsetupforoptimizationofelectromagnetic shieldingefficient



↓	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11
	Palladium Chloride	Nickel (II) Sulphate	Time	Temp.	R1	R2	R3	R4		SNRA1	MEAN1
3	17.5	876.2	20	50	34	30	31	32		30.0073	31.75
4	29.2	642.6	10	50	36	33	34	35		30.7427	34.50
5	29.2	759.4	20	30	37	34	35	36		30.9916	35.50
6	29.2	876.2	5	40	41	38	39	40		31.9215	39.50
7	40.9	642.6	20	40	45	42	43	44		32.7612	43.50
8	40.9	759.4	5	50	42	39	40	41		32.1392	40.50
9	40.9	876.2	10	30	44	40	41	43		32.4465	42.00

Figure-2- Experimental Design and Output

Step7:Conducting Experimentation.

1 Resultsand Discussion

Highershieldingeffectivenessvalueindicatesthebetterelectromagneticshieldingeffectivenessfromnickelplatedcoppercorefabricandhencelarger-the

-betterS/Nisemployedtooptimizetheprocessparameterofelectrolessnickelplating.

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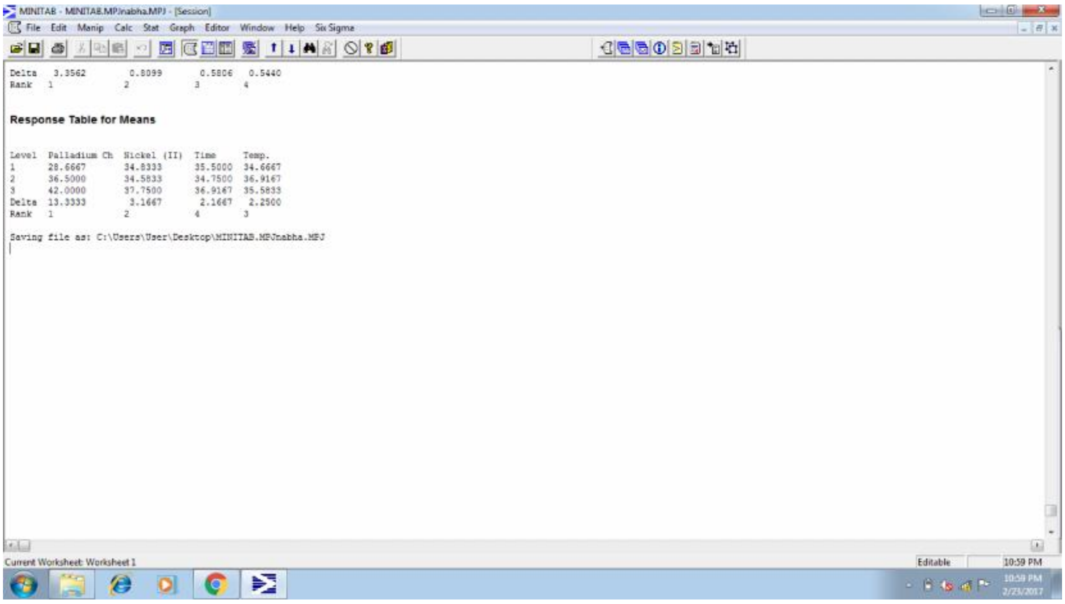
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Delta	3.3562	0.8099	0.5806	0.5440
Rank	1	2	3	4

Response Table for Means

Level	Palladium Ch	Nickel (II)	Time	Temp.
1	28.6667	34.8333	35.5000	34.6667
2	36.5000	34.5833	34.7500	36.9167
3	42.0000	37.7500	36.9167	35.5833
Delta	13.3333	3.1667	2.1667	2.2500
Rank	1	2	4	3

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Figure-3- Response table for means

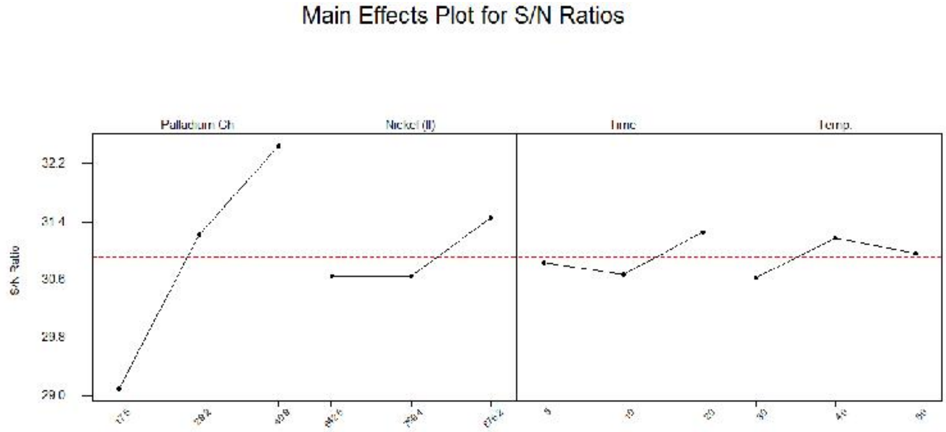

Figure 4- Main effects to plot an s/n ratio

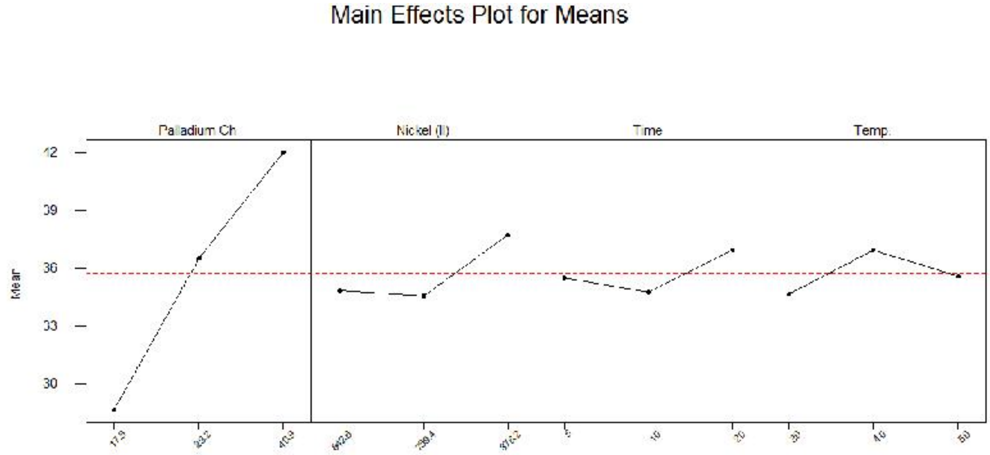

Figure-5-Main effects to plot mean ratio

Step 9: Prediction of the Expected Results

Table-2- S/N ratio and mean

S/N Ratio	Mean
31.57	38.17

Table-3- ANOVA

Source	Model DF	Reduced DF	Seq SS
PALLADIUM	2	2	67.837
NICKEL	2	2	57.065
TIME	2	2	117.502
TEMPERAT	2	0+	0
Error	17	19	216.25
Total	25	25	458.654

ANOVAcarriedoutforthesignal-to-noiseratioofvariouscombinationsofthedesignandnoisefactorsshowsthepredominantcontrollingnatureofpalladium chloride,nickel(II)sulphate,timeandtemperature.ANOVAresultsalsoshowthedominatingeffectsofpalladiumchlorideascomparedtonickel(II)sulphate,timeandtemperature.Effectofindividualvariablesintermsof theiraverageS/Nratioalongwiththe%factorseffectsandFvalues(pooledandunpooled)furtherconfirmthedominantnature.

ConfirmationtestsintheTaguchimethodsupplementsassuresthevalidityoftheresultsobtainedin theexperimentaldesigns,orthogonalarray selectedinthestudyandvariouslevelsofthedesignparametersandtheirinteractions.Confirmationtestcarriedoutwithoptimumparametersfortheresponsevariableshowsthecloserresultstothatoforiginalresultsanddoesnotshowanysignificantdifferenceat95%confidencelevels.Thevaluesobtainedinconfirmationtestsalongwiththeoriginalvaluesforresponsevariables,consideredinthestudy

Step 10: Validation of Optimal Setting

Since this is a study project therefore validation step will not be carried out

5. Conclusion

The Taguchi’s approach based is applied to Electroless nickel plated composite textile materials for electromagnet compatibility has been carried out for optimizing the performance. The various input parameters of the model have been optimized factors using SNR. The higher-the-better quality characteristic has been used for maximizing the electromagnet compatibility.

- Withanincrease in thepalladiumchlorideandnickel(II)sulphate,anincreaseinnickeldepositionandelectromagneticshieldinghasbeenobservedin200 -1000 MHzfrequencyrange.
- TheANOVAcarriedoutforEMSEofelectrolessnickelplatedcoppercore /polyestersheathyarncompositefabricshowstheneutral/negligibleeffects ontime andtemperaturefactors.

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