Process Control Plan for
PVC Resin[1] Moisture Content Control

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Summary:

The moisture content of PVC resin (powder) is an important factor for PVC grading. The better the grade of PVC resin can ensure the higher the profit for the manufacturer. The quality control of the moisture content is thus important along with other factors for example molecular weight, porosity, bulk density, and particle size distribution(PSD). The reason this moisture content analysis is selected in this study is to show its potential to be an on-line real time measurement. For laboratory analysis, usually a traditional microwave oven with a scale is the main equipment for the moisture measurement of PVC resin until the use of NIR (Near Infrared) spectroscopy as its potential replacement. The (simulated) data collected is based on the measurement from this NIR on PVC resin sampled every 3 hours for laboratory analysis. The process quality metrics is defined based on this equipment, the data collection is manual and the data can be saved in a computer that makes the creation of the control chart plan much easier. More data needed to be collected for the on-line conversion. Since NIR spectroscopy is portable, we recommend a designate team to collect the sample and analyze the sample right in the dryer area every 30 minutes for a prelude the on-line NIR measurements that links the on-line to real time process control of PVC moisture quality control. The X/R chart is very useful for the process monitoring and quality control in the PVC moisture measurements.

Introduction:

There are three main processes can be used for the commercial production of PVC powders 1) Suspension 2) Emulsion 3) Bulk methods. The PVC produced by suspension process provides 80% of worldwide support. So, the process of PVC polymerization will be focused on the suspension method. In this study, the Chisso[2] Process is used to produce PVC from vinyl chloride monomer (VCM) using suspension polymerization. The Chisso process sequences are illustrated in Figure 1.

PVC Suspension Process:

This process can be divided into 6 different steps from input of raw materials to the end products:

a) Input Fresh VCM, additives and water into a stirring reactor (1), and maintaining temperature during the polymerization to control the grade of the PVC

b) Discharge PVC powder after 85-90% VCM/PVC conversion to a blowdown tank (2) to flush off VCM gas and recover VCM gas to VCM gas holder (6)

c) PCV slurry containing VCM is fed into the stripping column (3) continuously, most of the residual VCM will be recovered from this column

d) The slurry will be de-watered with a centrifuge device (4)

e) The slurry will be dried by the proprietary dryer (5). It is then passed to storage silos.

PVC Process Inputs and Outputs:

Block diagram and process sequences can be seen from figure 1. This figure also identifies several needed raw materials and equipment for PVC polymerization. Many more inputs are needed to produce PVC resins.

Inputs:

- Capacity of the Reactor: 130 m3
- Mixing speed with rotator : 120 r/s
- Water Temperature in Jacket : 60 ^{o}C
- Reaction time (with 85% conversion) : 6 hours
- Raw Materials:
  1) VCM
  2) Purified Water
  3) Additives
     a) PVC additives
     b) Initiators
     c) Inhibitors
- Centrifuge Time: 10 minutes
- Drying Time: 2 Hours

Outputs:

- PVC powders with different grading.

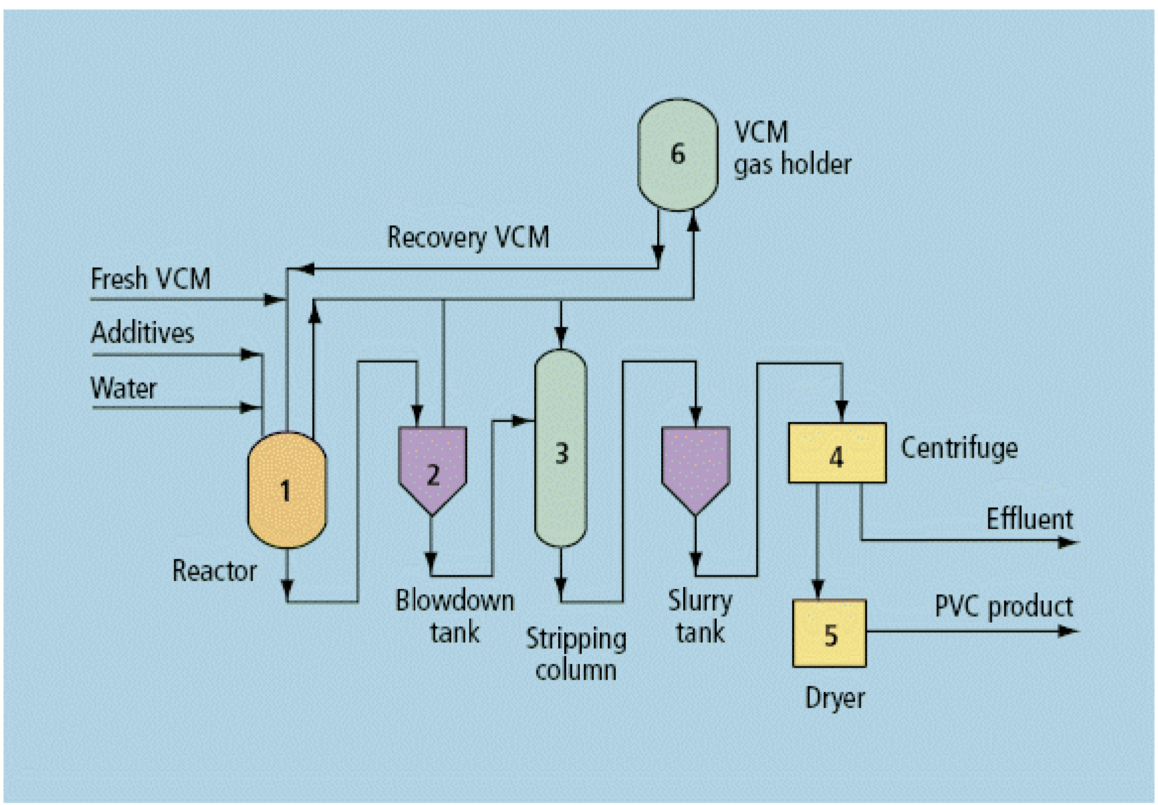

Figure 1 The Chisso Process for PVC suspension polymerization.

Process quality[3]/defect metrics on moisture contents:

For each grade of PVC resin, the plant applies a defined control plan which checks all necessary factors that impact on the PVC resin moisture content in order to guarantee the moisture content of specific grade upon delivery.

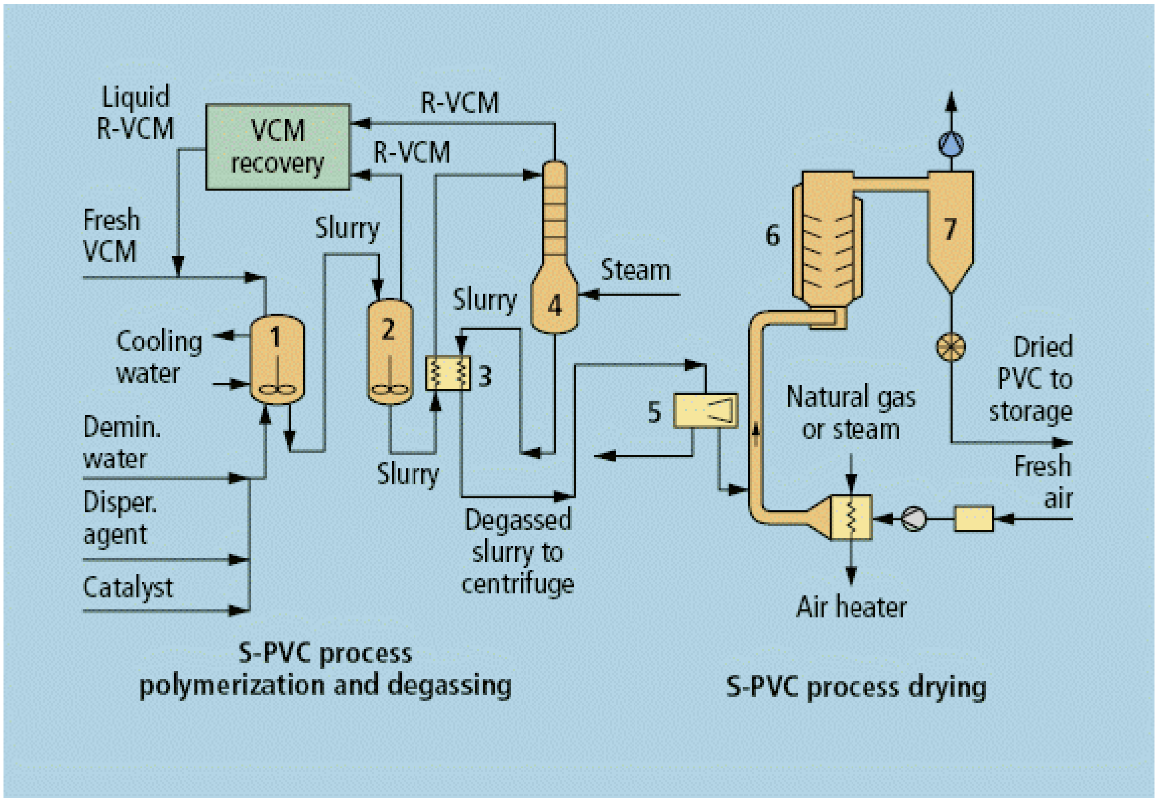

Figure 2 Production of suspension PVC resins and drying process using the Vinnolit[2] process.

These factors which are regularly controlled for drying purpose are:
• Stream pressure that regulates the temperature for drying
• Stream flow that regulates the temperature for drying
• Centrifuge speed
• RPM of dryer
• Speed of wet PVC slurry delivered to the dryer

PVC Resin quality control:

Root Cause analysis of potential defects as shown in figure 3 is the fishbone diagram that provides a cause-effect diagram for moisture control of PVC products.

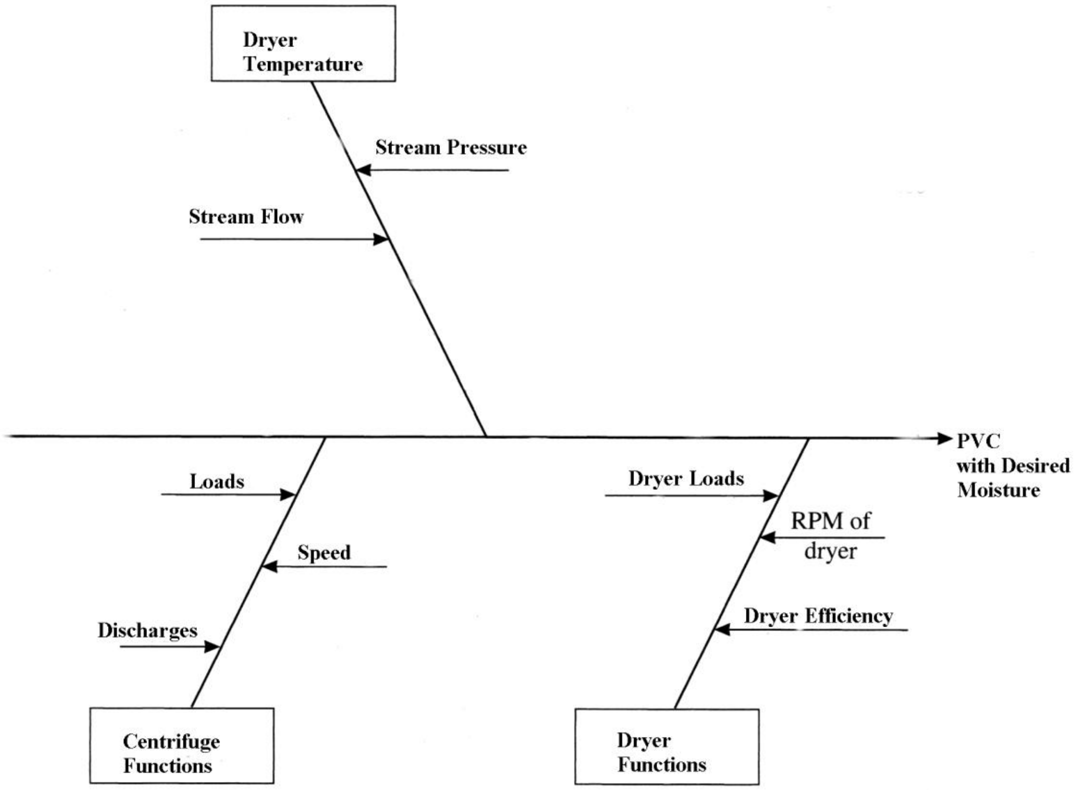

Figure 3 Fishbone diagram for PVC powder moisture control cause-effect factors.

Plan for quality Control

Quality Categories	Moisture Content
Grade A	0.08% max 0.06% min
Grade B	0.2% max 0.16% min
Grade C	0.4% max 0.32% min

Table 1 Grade[4] differences with different moisture contents.

There are many PVC grade defined based on PSD, Molecular weight, and moisture contents as well as other factors. This control plan focuses on the drying process of the PVC manufacturing process. The moisture content in PVC becomes the key quality factor in this process. Several different grades of PVC resin based on the content of moisture are listed in table 1. The outcomes of these different grades are the combination effects of drying process attributes such as 1) Dryer Functional Capacities 2) Dryer's Temperature 3) Centrifuge Functional Capacities as outlined in figure 3.

Data Collection Plan:

The data collection process is quite straight forward. The dried samples were collected very 3 hours from the dryer then delivered to the laboratory for analysis. The samples collected will be divided into 5 sub-groups and measured using NIR spectroscopy.

Time Collected	sample 1	sample 2	sample 3	sample 4	sample 5
1/8/2008 0:00	0.052502	0.065885	0.072704	0.07729	0.055188
1/8/2008 3:00	0.046729	0.07814	0.07503	0.072986	0.057855
1/8/2008 6:00	0.081042	0.061769	0.07383	0.079706	0.042587
1/8/2008 9:00	0.066987	0.054687	0.073257	0.079485	0.031263
1/8/2008 12:00	0.054148	0.075275	0.075287	0.070181	0.06914
1/8/2008 15:00	0.051889	0.077382	0.062814	0.079918	0.072422
1/8/2008 18:00	0.041749	0.061305	0.072909	0.077687	0.074892
1/8/2008 21:00	0.065373	0.053532	0.079079	0.079398	0.03872
1/9/2008 0:00	0.056421	0.070201	0.064231	0.070914	0.057024
1/9/2008 3:00	0.041434	0.074895	0.065569	0.073827	0.073755
1/9/2008 6:00	0.067223	0.067477	0.060618	0.071289	0.060789
1/9/2008 9:00	0.043065	0.077134	0.069552	0.07658	0.056155
1/9/2008 12:00	0.067645	0.061688	0.064755	0.072056	0.063425
1/9/2008 15:00	0.08155	0.062856	0.063601	0.078277	0.039625
1/9/2008 18:00	0.076182	0.073764	0.078999	0.072229	0.056922
1/9/2008 21:00	0.049503	0.069467	0.075433	0.070205	0.073062
1/10/2008 0:00	0.047152	0.054716	0.067374	0.071579	0.060499
1/10/2008 3:00	0.069627	0.075459	0.072682	0.073338	0.049903
1/10/2008 6:00	0.06023	0.07446	0.071719	0.078592	0.074355
1/10/2008 9:00	0.059426	0.053129	0.06013	0.070254	0.058478
1/10/2008 12:00	0.044073	0.060969	0.065462	0.072211	0.044099
1/10/2008 15:00	0.080496	0.077109	0.068689	0.071218	0.075765
1/10/2008 18:00	0.040792	0.061931	0.076944	0.070996	0.033552
1/10/2008 21:00	0.064018	0.073495	0.073682	0.072554	0.059126
1/11/2008 0:00	0.054878	0.077266	0.073198	0.078431	0.049399
1/11/2008 3:00	0.063761	0.07029	0.065626	0.071621	0.030612
1/11/2008 6:00	0.046135	0.053693	0.063016	0.075978	0.063
1/11/2008 9:00	0.07028	0.053777	0.078986	0.077811	0.077528
1/11/2008 12:00	0.077827	0.069619	0.075164	0.078864	0.057862
1/11/2008 15:00	0.067903	0.066594	0.071089	0.07515	0.061918
1/11/2008 18:00	0.048657	0.056714	0.077467	0.077499	0.052693
1/11/2008 21:00	0.042619	0.07974	0.0711	0.07597	0.064295
1/12/2008 0:00	0.050472	0.070069	0.064526	0.078627	0.077009
1/12/2008 3:00	0.045763	0.065038	0.075645	0.078275	0.053688
1/12/2008 6:00	0.062615	0.056947	0.068779	0.076805	0.077084
1/12/2008 9:00	0.065153	0.071225	0.073238	0.071075	0.066181
1/12/2008 12:00	0.047358	0.068896	0.061143	0.073592	0.068008
1/12/2008 15:00	0.052853	0.050715	0.069304	0.07988	0.064999
1/12/2008 18:00	0.082044	0.066452	0.070319	0.079316	0.031494
1/12/2008 21:00	0.060719	0.055909	0.069736	0.076738	0.032901

Table 2 the measured moisture data (simulated) for PVC resin samples
The value of the sample is % weight of moisture per sample weight.

NIR spectroscopy[5]:

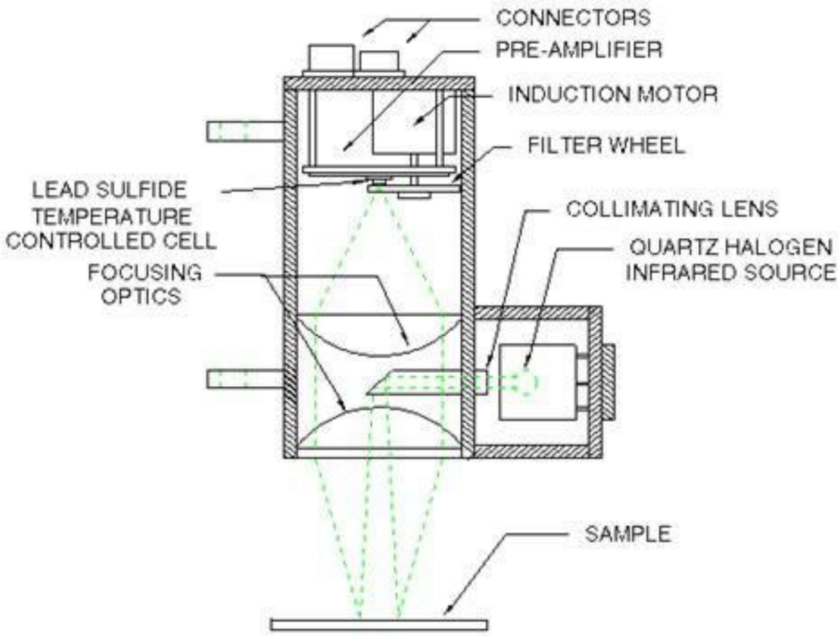

Figure 4 A typical NIR equipment for moisture measurements.

The NIR spectroscopy can measure the moisture contents of PVC from 0% to 75%. The calibration can be performed using standards from NIST.

Control Chart Plan:

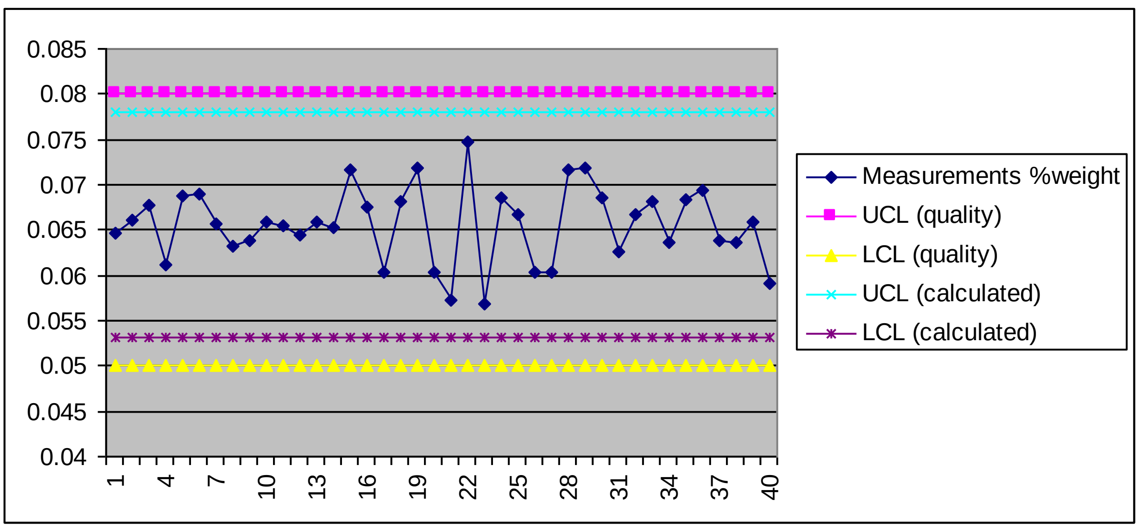

Figure 5 PVC moisture content from dryer from 01/01/08 to 01/12/08.

For this PVC grade, the specification for the moisture contents are between 0.05% to 0.08%. With the 40 samples of 5 measurement each, we could obtained the statistics and calculated the UCL and LCL. The statistics of the measurement are in table 2.

Column1

Mean	0.065631618
Standard Error	0.0006541
Median	0.065904901
Mode	#N/A
Standard Deviation	0.004136892
Sample Variance	1.71139E-05
Kurtosis	-0.270538296
Skewness	-0.142469321
Range	0.017812244
Minimum	0.0568431
Maximum	0.074655345
Sum	2.625264718
Count	40

Table 2 statistics of measurements

So, the UCL = 0.0656 + 3 * 0.00413 = 0.0780

LCL = 0.0656 – 3 * 0.00413 = 0.0532

Figure 5 shows the measurement from 01/01/08 to 01/12/08. The criteria of process in control are a) No sample points outside of process limits b) Most points near average c) Nearly equal number of point above and below average d) Points are randomly distributed. Use these criteria, the process should be in control.

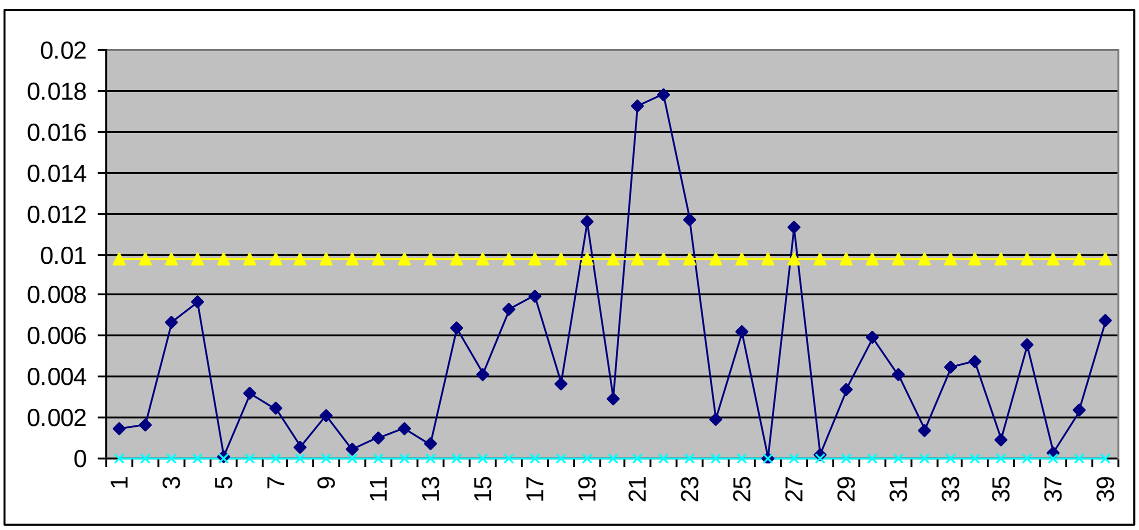

Figure 6 R-bar chart of PVC moisture content from dryer from 01/01/08 to 01/12/08.

The UCL of range-chart is D2 * R-bar = 0.00974

The LCL of range-chart is D3 * R-bar = 0

Figure 6 shows the point 19, 21, 22, 23 (measurements on 01/10/08) are out of ucl limit, this is the evidence that the uniformity of the process has changed. These out of control happened in 01/10/08, so it is easy to track what happened to these measurements (control) by checking the changing of personnel on measurements, or changing of attributes that impacts the drying efficiency.

Conclusion:

PVC resin drying technology is now quite mature and seems little left to improve. But with the NIR measurements of moisture content that provides a means for potential on-line process control. The control chart of X/R chart on the off line measurements show how the measurements can link to the proper identifications of attributes that contributes the process to be out of control. By combining the cause-effect diagram, this off-line process control can be converted to an on-line NIR process measurement. This on-line process control will eventually have the feed-back loop that correct the attributes that upset the process from the real time X/R chart. The purpose of this study is to show the possibility of an on-line NIR moistures measurements for PVC dryer process control.

References:

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5. How NIR works, retreated from http://www.moistureregisterproducts.com/faq.htm on August 8, 2008